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APPENDIX A

STANDARD OPERATING PROCEDURES FOR NON-CLP ANALYTICAL METHODS

HAUSER

HAUSER CHEMICAL RESEARCH, INC.

VISCOSITY-KINEMATIC Standart test method ASTM D445-86
Annual book of ASTM standards section 5.01.

HAUSER LABORATORIES
A Division

SCOPE: This test method covers the determination of the kinematic viscosity of liquid petroleum products both transparent and opaque by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer.

DEFINITONS: Kinematic viscosity -- a measure of the resistive flow of a fluid under gravity, the pressure head being proportional to the density, p, of the fluid: for gravity flow under a given hydrostatic head, the pressure head of a liquid is proportional to its density,p. For any particulaar viscometer, the time of flow of a fixed volume of fluid is directly proportional to its kinematic viscosity, v = n/p, where n is the dynamic viscosity coefficient.

PROCEDURE: follow general procedure sections 8 and 9.

CALCULATION/RESULTS:

kinematic viscosity = cSt = Ct

where:

C = calibration constant
t = flow time

NOTES:
1. Use ASTM thermometers for viscometer baths (table 2). Maintain temperature +/- 0.02 F.
2. Use viscometers as described (table 1)
3. Between determinations thoroughly clean viscometers with appropriate solvents, and periodically clean with chromic acid. (section 12)
4. Viscosity standards: used as confirmatory checks on the procedure in the laboratory, and for redetermination of calibration constant.(ASTM D446).
5. Recalibration: based on use. Recalibrate every 10 runs or when standard varies by more than +/- 0.35%.
6. Run duplicate tests on 10% of samples.

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Designation: D 445 – 88

An American National Stand
British Standard 4

Designation: 71/84

Standard Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[1]

This standard is issued under the fixed designation D 445; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval. This is also a standard of the Institute of Petroleum issued under the fixed designation IP 71. The final number indicates the year of last revision.

This test method has been approved by the sponsoring committee and accepted by the Cooperating Societies in accordance with established procedures.

This standard has been approved for use by agencies of the Department of Defense and replaces Method 305.6 of Federal Test Method Standard 791b. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 This test method covers the determination of the kinematic viscosity of liquid petroleum products (Note 1), both transparent and opaque, by measuring the time for a volume of liquid to flow under gravity through a calibrated glass capillary viscometer. The dynamic viscosity can be obtained by multiplying the measured kinematic viscosity by the density of the liquid.

NOTE 1—For the measurement of the viscosity of bitumens, see also Test Method D 2170 and Test Method D 2171.

1.2 This test method is intended primarily for application to liquids for which the shear stress and shear rates are proportional.

1.2.1 This test method depends on the behavior of the sample, and ideally the coefficient of viscosity should be independent of the rate of shear (this is commonly called Newtonian flow behavior). If, however, the coefficient of viscosity varies significantly with the rate of shear, different results may be obtained from viscometers of different capillary diameters.

1.3 This test method also includes the determination of the kinematic viscosity of fuel oils which often exhibit non-Newtonian properties.

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 446 Specifications and Operating Instructions for Gl Capillary Kinematic Viscometers[2]

D 2170 Test Method for Kinematic Viscosity of Asphal (Bitumens)[3]

D 2171 Test Method for Viscosity of Asphalts by Vacuu Capillary Viscometer[3]

E 1 Specification for ASTM Thermometers[4]

E 77 Method for Inspection and Verification of Liqui in-Glass Thermometers[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *kinematic viscosity*—a measure of the resistive flo of a fluid under gravity, the pressure head being proportion to the density, ρ, of the fluid: for gravity flow under a giv hydrostatic head, the pressure head of a liquid is propo tional to its density, ρ. For any particular viscometer, t time of flow of a fixed volume of fluid is directly propo tional to its kinematic viscosity, $\nu = \eta/\rho$, where η is t dynamic viscosity coefficient. The kinematic viscosity coef cient has the dimension L^2/T, where L is a length, and T is time. The cgs unit of kinematic viscosity is one centimet squared per second and is called one stokes (symbol St). Th SI unit of kinematic viscosity is one metre squared p second and is equivalent to 10^4 St. Frequently, t centistokes (symbol cSt) is used (1 cSt = 10^{-2} St = 1 mm^2/s

3.1.2 *density*—the mass per unit volume of the fluid. Th dimension of density is M/L^3, where M is a mass. The c unit of density (ρ) is one gram per millilitre, and the SI un of density is one kilogram per cubic metre.

3.1.3 *dynamic viscosity* (coefficient of)—the ratio betwe the applied shear stress and rate of shear. This coefficient, is thus a measure of the resistance to flow of the fluid; it commonly called the viscosity of the liquid. The dimensio of the coefficient of dynamic viscosity is $M/LT = FT/L$

[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.07 on Flow Properties.

Current edition approved Oct. 31, 1988. Published December 1988. Originally published as D 445 – 37 T. Last previous edition D 445 – 86[ε1].

In the IP, this test method is under the jurisdiction of the Standardization Committee.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 04.03.

[4] *Annual Book of ASTM Standards*, Vols 05.01 and 14.03.

[5] *Annual Book of ASTM Standards*, Vol 14.01.

TABLE 1 Viscometer Types

Viscometer Identification	Range, cSt (mm^2/s)[A]
A Glass Types for Transparent Liquids:	
1. Cannon-Fenske Routine[C]	0.5[B] to 20 000
2. Zeitfuchs[C]	0.6 to 8 000
3. BS/IP/IS[C]	0.8 to 10 000
4. Cannon-Manning Semi-micro[C]	0.4 to 20 000
5. BS/IP U-tube[C]	0.9[B] to 19 000
6. BS/IP U-tube Miniature[C]	0.2 to 100
7. Pinkevitch	0.8[B] to 17 000
B Suspended-Level Types for Transparent Liquids:	
1. Ubbelohde[C]	0.3[B] to 100 000
2. PinSimons[C]	0.6 to 1 200
3. Atlantic[C]	0.75[B] to 5 000
4. Cannon-Ubbelohde, Cannon-Ubbelohde Dilution[C]	0.5[B] to 100 000
5. Cannon-Ubbelohde Semi-micro[C]	0.4 to 20 000
6. BS/IP Suspended Level[C]	3.5[B] to 100 000
7. BS/IP Suspended Level, Shortened Form[C]	1.05[B] to 10 000
8. BS/IP Miniature Suspended Level[C]	0.6 to 6 000
C Reverse-Flow Types for Transparent and Opaque Liquids:	
1. Zeitfuchs Cross-Arm[C]	0.6 to 100 000
2. Cannon-Fenske Opaque[C]	0.4 to 20 000
3. Lantz-Zeitfuchs[C]	60 to 100 000
4. BS/IP U-tube Reverse Flow[C]	0.6 to 300 000

[A] Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Table 4.

[B] In each of these series, the minimum flow time for the viscometers with the lowest constant exceeds 200 s.

[C] Specifications and operating instructions for these viscometers have been standardized in Specifications and Operating Instructions D 446.

[D] Specifications for these are given in Appendixes to IP 71.

depending on whether the dimension of viscosity is based on the *M-L-T* system or the *F-L-T* system (where *F* represents a force). The cgs unit of dynamic viscosity is one gram per centimetre per second = one dyne-second per centimetre squared and is called one poise (symbol P). Frequently, the centipoise (symbol cP) is used (1 cP = 10^{-2} P). The SI unit of dynamic viscosity is one pascal-second; for convenience its submultiple of millipascal-second is frequently used (1 mPa·s = 1 cP).

NOTE 2—Dynamic viscosity also denotes a frequency-dependent property in which shear stress and shear rate have a sinusoidal time dependence. It is hoped that this dual use of the same term will not be confusing.

4. Summary of Test Method

4.1 The time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

5. Significance and Use

5.1 Many petroleum products, as well as nonpetroleum materials, are used as lubricants for bearings, gears, compressor cylinders, hydraulic equipment, etc. The proper operation of the equipment depends upon the proper kinematic viscosity or viscosity (sometimes called dynamic viscosity) of the liquid. Thus, the accurate measurement of kinematic viscosity and viscosity is essential to many product specifications.

5.2 The kinematic viscosity of many petroleum fuels is important for their proper use, for example, flow of fuels through pipe lines, injection nozzles and orifices, and the determination of the temperature range for proper operation of the fuel in burners.

6. Apparatus

6.1 *Viscometers* of the glass capillary type, calibrated and capable of measuring kinematic viscosity within the limits of precision given in Section 15 are acceptable. Viscometers listed in Table 1 meet these requirements.

6.1.1 Automated assemblies that measure kinematic viscosity within the limits of precision given in Section 15 are acceptable alternatives; kinematic viscosities less than 10 cSt (mm^2/s) and flow times less than 200 s may require a kinetic energy correction (see Specification D 446).

6.2 *Viscometer Holders* to enable the viscometer to be suspended in a similar position as when calibrated. The proper alignment of vertical parts may be confirmed by using a plumb line.

6.3 *Viscometer Thermostat and Bath*—Any transparent liquid or vapor bath may be used, provided that it is of sufficient depth that at no time during the measurement will any portion of the sample in the viscometer be less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 The temperature control must be such that for the range from 15 to 100°C (60 to 212°F) the temperature of the bath medium does not vary by more than 0.01°C (0.02°F) over the length of the viscometers, or between the position of each viscometer, or at the location of the thermometer. For

TABLE 2 Kinematic Viscosity Test Thermometers[A]

Test Temperature[B]	Scale Error[B]	Thermometer Number	
°F	°C	ASTM[C]	IP[D]
−65	−53.9	74F, Ct†	66F, C
−60 to −65	−51 to −53	42F	62F, C
−40	−40	73F, C	66F, C
−15	−26.1	126F, C	71F, C
	−20	127C	20C
0	−17.8	72F, Ct†	67F, C
32	0	128F, C	59F, C
68 and 70	20 and 21.1	44F, C	23F, C
77	25	45F, C	30F, C
86	30	118F, C	
100	37.8	28F, C	31F, C
	40	1200	32C
122	50	46F, C	68F, C
130	54.4	29F, Ct†	34F, C
140	60	47F, C	35F, C
	80		100C
160	69.9	48F, C	90F, C
200	93.3	129F, C	39F, C
210 and 212	98.9 and 100	50F	38F, C
	100	121C	
275	135	110F, C	

[A] The smallest graduation of the Fahrenheit thermometers is 0.1°F and for the Celsius thermometers is 0.05°C except for ASTM 49F and IP 65F for which it is 0.2°F.

[B] Scale error for the Fahrenheit thermometers is not to exceed ±0.2°F (except for ASTM 110F which is ±0.3°F); for the Celsius thermometers it is ±0.1°C. These scale errors are required to apply only at the given test temperature.

[C] Complete construction detail is given in Specification E 1.

[D] Complete construction detail is given in Part I of IP Standards for Petroleum and Its Products.

† Stemmary corrected.

TABLE 3 Values of the Several ASTM Viscosity Oil Standards

Viscosity Standard Conforming to ASTM Standards[A]	Approximate Kinematic Viscosity, cSt (mm²/s)							
	At −40°C (−40°F)	At 20°C (68°F)	At 25°C (77°F)	At[B] 37.78°C (100°F)	At 40°C (104°F)	At 50°C (122°F)	At[B] 98.89°C (210°F)	At 100 (212
S-3	90	4.6	4.0	3.6	2.9		1.2	1.2
S-6		11	8.8	6.0	5.7		1.8	1.8
S-20		44	34	28	18		4.0	3.9
S-60		170	130	95	54		7.4	7.2
S-200		840	400	200	180		17	17
S-600		2400	1600	900	520	280	35	32
S-2000		8700	5600	2500	1700		78	76
S-6000		37 000	26 000	8000	6700			1
S-30 000			81 000	27 000	23 000	11 000		

[A] The actual values for the standards listed above are established and annually reaffirmed by cooperative tests. In 1985 tests were made using 16 different types viscometers in 26 laboratories located in 13 countries.

[B] Standardizations at 37.78°C and 98.89°C may be discontinued January 1, 1987.

temperatures outside this range, the variation must not exceed 0.03°C (0.05°F).

6.4 *Temperature-Measuring Device*—Standardized liquid-in-glass thermometers (Table 3) of an accuracy after correction of 0.02°C (0.04°F) can be used, or any other thermometric device of equal or better accuracy. If standardized liquid-in-glass thermometers are used, it is recommended (but not required) that two thermometers be used; they must agree within 0.04°C (0.07°F).

6.5 *Timing Device*—Any timing device may be used provided that the readings can be taken with a discrimination of 0.2 s or better, and that it has an accuracy within ±0.07 % when tested over intervals of 15 min.

6.5.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in viscosity flow measurements.

7. Reagents and Materials

7.1 *Appropriate Solvent*, completely miscible with the sample, for example, petroleum ether.

7.2 *Appropriate Volatile Solvent*, completely miscible with the solvent described in 7.1.

7.3 *Chromic Acid Solution* for cleaning glassware.

8. Calibration

8.1 *Viscometers*—Use only calibrated viscometers with constants measured and provided to the nearest 0.1 % of their value.

8.2 *Thermometers*—Routine liquid-in-glass thermometers should be checked to the nearest 0.01°C (0.02°F) by direct comparison with a suitable calibrated thermometer.

TABLE 4 Minimum Flow Times

Note—All sizes of all viscometers listed in Specification D 446 are designed for a flow-time in excess of 200 s, except as noted below. The minimum flow times for the six "88/U" viscometers listed in Table 1 are given in the appendices to IP71.

Viscometer Identification	ASTM Size	Minimum Flow-Time, s
Cannon-Fenske routine	25	200
Ubbelohde	0	300
Atlantic	OC	250
Cannon-Ubbelohde, Cannon-Ubbelohde dilution	25	200

8.2.1 Kinematic viscosity test thermometers shall be standardized at total immersion which means immersion to the top of the mercury column, with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature. Do not submerge the expansion bulb at the top of the thermometer.

8.2.2 It is essential that the ice point of standardized thermometers be determined periodically and that the [illegible] cial corrections be adjusted to conform to the change in point.

8.3 *Timers*—Standard time signals available in some countries may be used for checking accuracy of timing devices.

8.4 *Viscosity Standards*[A] (Table 3)—These may be used as confirmatory checks on the procedure in the laboratory. If the measured kinematic viscosity does not agree within ±0.35 % of the certified value, each step in the procedure should be rechecked, including thermometer and viscometer calibration, to locate the source of error. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

8.4.1 *Viscosity Oil Standards*: ASTM, having the approximate kinematic viscosity shown in Table 3 are available. Certified kinematic viscosity values are compared by annual cooperative tests by a number of laboratories. The current values are supplied with each portion.

9. General Procedure for Kinematic Viscosity

9.1 The specific details of operation vary for the different types of viscometers listed in Table 1. The operating instructions for the different types of viscometers are given in Specification D 446.

9.2 Maintain the bath at the test temperature within the limits given in 6.3.1 taking account of the precautions given in Appendix X1 and of the correction supplied on the certificates of calibration.

9.2.1 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used. The thermometers should be held in an upright position under the same

[A] The ASTM Viscosity Oil Standards are available in 1-qt (0.47 L) containers. Purchase orders should be addressed to the Cannon Instrument Co., P.O. Box 16, State College, PA 16801. Shipment will be made as specified or by best means.

TABLE 1 Viscometer Types

Viscometer Identification	Range, cSt (mm^2/s)[A]
1 Ostwald Types for Transparent Liquids:	
1. Cannon Fenske Routine[C]	0.5[B] to 20 000
2. Zeitfuchs[C]	0.6 to 3 000
3. SIL[C]	0.6 to 10 000
4. Cannon-Manning Semi-micro[C]	0.4 to 20 000
5. BS/IP U-tube[D]	0.9[B] to 10 000
6. BS/IP U-tube Miniature[D]	0.2 to 100
7. Pinkevitch	0.6[B] to 17 000
3 Suspended-Level Types for Transparent Liquids:	
1. Ubbelohde[C]	0.3[B] to 100 000
2. FitzSimons[C]	0.6 to 1 200
3. Atlantic[C]	0.75[B] to 5 000
4. Cannon-Ubbelohde, Cannon-Ubbelohde Dilution[C]	0.5[B] to 100 000
5. Cannon-Ubbelohde Semi-micro[C]	0.4 to 20 000
6. BS/IP Suspended Level[D]	3.5[B] to 100 000
7. BS/IP Suspended Level, Shortened Form[D]	1.05[B] to 10 000
8 BS/IP Miniature Suspended Level[D]	0.6 to 3 000
2 Reverse-Flow Types for Transparent and Opaque Liquids:	
1. Zeitfuchs Cross-Arm[C]	0.6 to 100 000
2. Cannon-Fenske Opaque[C]	0.4 to 20 000
3. Lantz-Zeitfuchs[C]	80 to 100 000
4. BS/IP U-tube Reverse Flow[D]	0.6 to 300 000

[A] Each range quoted requires a series of viscometers. To avoid the necessity of making a kinetic energy correction, these viscometers are designed for a flow time in excess of 200 s except where noted in Table 4.

[B] In each of these series, the minimum flow time for the viscometers with the lowest constant exceeds 200 s.

[C] Specifications and operating instructions for these viscometers have been assembled in Specifications and Operating Instructions D 446.

[D] Specifications for these are given in Appendixes to IP 71.

depending on whether the dimension of viscosity is based on the *M-L-T* system or the *F-L-T* system (where *F* represents a force). The cgs unit of dynamic viscosity is one gram per centimetre per second = one dyne-second per centimetre squared and is called one poise (symbol P). Frequently, the centipoise (symbol cP) is used (1 cP = 10^{-2} P). The SI unit of dynamic viscosity is one pascal-second; for convenience its submultiple of millipascal-second is frequently used (1 mPa·s = 1 cP).

NOTE 2—Dynamic viscosity also denotes a frequency-dependent quantity in which shear stress and shear rate have a sinusoidal time dependence; it is hoped that this dual use of the same term will not be confusing.

4. Summary of Test Method

4.1 The time is measured in seconds for a fixed volume of liquid to flow under gravity through the capillary of a calibrated viscometer under a reproducible driving head and at a closely controlled temperature. The kinematic viscosity is the product of the measured flow time and the calibration constant of the viscometer.

5. Significance and Use

5.1 Many petroleum products, as well as nonpetroleum materials, are used as lubricants for bearings, gears, compressor cylinders, hydraulic equipment, etc. The proper operation of the equipment depends upon the proper kinematic viscosity or viscosity (sometimes called dynamic viscosity) of the liquid. Thus, the accurate measurement of kinematic viscosity and viscosity is essential to many product specifications.

5.2 The kinematic viscosity of many petroleum fuels is important for their proper use, for example, flow of fuels through pipe lines, injection nozzles and orifices, and the determination of the temperature range for proper operation of the fuel in burners.

6. Apparatus

6.1 *Viscometers* of the glass capillary type, calibrated and capable of measuring kinematic viscosity within the limits of precision given in Section 15 are acceptable. Viscometers listed in Table 1 meet these requirements.

6.1.1 Automated assemblies that measure kinematic viscosity within the limits of precision given in Section 15 are acceptable alternatives; kinematic viscosities less than 10 cSt (mm^2/s) and flow times less than 200 s may require a kinetic energy correction (see Specification D 446).

6.2 *Viscometer Holders* to enable the viscometer to be suspended in a similar position as when calibrated. The proper alignment of vertical parts may be confirmed by using a plumb line.

6.3 *Viscometer Thermostat and Bath*—Any transparent liquid or vapor bath may be used, provided that it is of sufficient depth that at no time during the measurement will any portion of the sample in the viscometer be less than 20 mm below the surface of the bath liquid or less than 20 mm above the bottom of the bath.

6.3.1 The temperature control must be such that for the range from 15 to 100°C (60 to 212°F) the temperature of the bath medium does not vary by more than 0.01°C (0.02°F) over the length of the viscometers, or between the position of each viscometer, or at the location of the thermometer. For

TABLE 2 Kinematic Viscosity Test Thermometers[A]

Test Temperature[B]	Scale Error[B]	Thermometer Number	
°F	°C	ASTM[C]	IP[D]
−65	−53.9	74F, C†	69F, C
−60 to −35	−51 to −35	43F	65F, C
−40	−40	73F, C	68F, C
−15	−26.1	126F, C	71F, C
	−20	127C	99C
0	−17.8	72F, C†	67F, C
32	0	128F, C	33F, C
60 and 70	20 and 21.1	44F, C	29F, C
77	25	45F, C	30F, C
86	30	110F, C	
100	37.8	28F, C	31F, C
	40	129C	92C
122	50	46F, C	66F, C
130	54.4	29F, C†	34F, C
140	60	47F, C	35F, C
	80		100C
180	82.2	48F, C	90F, C
200	93.3	130F, C	36F, C
210 and 212	98.9 and 100	30F	32F, C
	100	131C	
275	135	110F, C	

[A] The smallest graduation of the Fahrenheit thermometers is 0.1°F and for the Celsius thermometers is 0.05°C except for ASTM 43F and IP 65F for which it is 0.2°F.

[B] Scale error for the Fahrenheit thermometers is not to exceed ±0.2°F (except for ASTM 110F which is ±0.3°F); for the Celsius thermometers it is ±0.1°C. These scale errors are required to apply only at the given test temperature.

[C] Complete construction detail is given in Specification E 1.

[D] Complete construction detail is given in Part I of IP Standards for Petroleum and its Products.

† Editorially corrected.

TABLE 2 Values of the Several ASTM Viscosity Oil Standards

Viscosity Standard Conforming to ASTM Standards[A]	Approximate Kinematic Viscosity, cSt (mm²/s)							
	At −40°C (−40°F)	At 20°C (68°F)	At 25°C (77°F)	At[B] 37.78°C (100°F)	At 40°C (104°F)	At 50°C (122°F)	At[B] 98.89°C (210°F)	At 100°C (212°F)
S-3	80	4.6	4.0	3.0	2.9		1.2	1.2
S-6		11	8.9	6.0	5.7		1.8	1.8
S-20		44	34	20	18		4.0	3.9
S-60		170	120	60	54		7.4	7.2
S-200		640	450	200	180		17	17
S-600		2400	1600	600	520	280	33	32
S-2000		8700	5600	2000	1700		78	75
S-8000		37 000	23 000	8000	6700			
S-30 000			81 000	27 000	23 000	11 000		

[A] The actual values for the standards listed above are established and annually reaffirmed by cooperative tests. In 1985 tests were made using 16 different types of viscometers in 28 laboratories located in 13 countries.

[B] Standardizations at 37.78°C and 98.89°C may be discontinued January 1, 1987.

temperatures outside this range, the variation must not exceed 0.03°C (0.05°F).

6.4 *Temperature-Measuring Device*—Standardized liquid-in-glass thermometers (Table 2) of an accuracy after correction of 0.02°C (0.04°F) can be used, or any other thermometric device of equal or better accuracy. If standardized liquid-in-glass thermometers are used, it is recommended (but not required) that two thermometers be used; they must agree within 0.04°C (0.07°F).

6.5 *Timing Device*—Any timing device may be used provided that the readings can be taken with a discrimination of 0.2 s or better, and that it has an accuracy within ±0.07 % when tested over intervals of 15 min.

6.5.1 Electrical timing devices may be used if the current frequency is controlled to an accuracy of 0.05 % or better. Alternating currents, as provided by some public power systems, are intermittently rather than continuously controlled. When used to actuate electrical timing devices, such control can cause large errors in viscosity flow measurements.

7. Reagents and Materials

7.1 *Appropriate Solvent*, completely miscible with the sample, for example, petroleum ether.

7.2 *Appropriate Volatile Solvent*, completely miscible with the solvent described in 7.1.

7.3 *Chromic Acid Solution* for cleaning glassware.

8. Calibration

8.1 *Viscometers*—Use only calibrated viscometers with constants measured and provided to the nearest 0.1 % of their value.

8.2 *Thermometers*—Routine liquid-in-glass thermometers should be checked to the nearest 0.01°C (0.02°F) by direct comparison with a suitable calibrated thermometer.

TABLE 4 Minimum Flow Times

NOTE—All sizes of all viscometers listed in Specification D 446 are designed for a flow-time in excess of 200 s, except as listed below. The minimum flow times for the six 'BS/IP' viscometers listed in Table 1 are given in the appendices to IP71.

Viscometer Identification	ASTM Size	Minimum Flow-Time, s
Cannon-Fenske routine	25	250
Ubbelohde	0	300
Atlantic	0C	250
Cannon-Ubbelohde, Cannon-Ubbelohde dilution	25	250

8.2.1 Kinematic viscosity test thermometers shall be standardized at total immersion which means immersion to the top of the mercury column, with the remainder of the stem and the expansion chamber at the top of the thermometer exposed to room temperature. Do not submerge the expansion bulb at the top of the thermometer.

8.2.2 It is essential that the ice point of standardized thermometers be determined periodically and that the official corrections be adjusted to conform to the change in ice point.

8.3 *Timers*—Standard time signals available in some countries may be used for checking accuracy of timing devices.

8.4 *Viscosity Standards*[6] (Table 3)—These may be used as confirmatory checks on the procedure in the laboratory. If the measured kinematic viscosity does not agree within ±0.35 % of the certified value, each step in the procedure should be rechecked, including thermometer and viscometer calibration, to locate the source of error. It must be appreciated that a correct result obtained on a standard oil does not preclude the possibility of a counterbalancing combination of the possible sources of error.

8.4.1 *Viscosity Oil Standards*, ASTM, having the approximate kinematic viscosity shown in Table 3 are available.[6] Certified kinematic viscosity values are compared by annual cooperative tests by a number of laboratories. The current values are supplied with each portion.

9. General Procedure for Kinematic Viscosity

9.1 The specific details of operation vary for the different types of viscometers listed in Table 1. The operating instructions for the different types of viscometers are given in Specification D 446.

9.2 Maintain the bath at the test temperature within the limits given in 6.3.1 taking account of the precautions given in Appendix X1 and of the correction supplied on the certificates of calibration.

9.2.1 In order to obtain the most reliable temperature measurement, it is recommended that two thermometers with valid calibration certificates be used. The thermometers should be held in an upright position under the same

[6] The ASTM Viscosity Oil Standards are available in 1-pt (0.47 L) containers. Purchase orders should be addressed to the Cannon Instrument Co., P.O. Box 16, State College, PA. 16804. Shipment will be made as specified or by best means.

conditions of immersion as when calibrated. They should be viewed with a lens assembly giving about five times magnification and which should be arranged to eliminate parallax errors.

9.3 Select a clean dry, calibrated viscometer having a range covering the estimated kinematic viscosity (that is, a wide capillary for a very viscous liquid and a narrower capillary for a more fluid liquid). The flow time should not be less than 200 s, or as noted in Table 4.

9.3.1 When the test temperature is below the dew point, affix loosely packed drying tubes to the open ends of the viscometer. The drying tubes must fit the design of the viscometer and not restrict the flow of the sample by pressures created in the instrument. Carefully flush the moist room air from the viscometer by applying vacuum to one of the drying tubes. Finally, before placing the viscometer in the bath, draw up the sample into the working capillary and timing bulb and allow to drain back as an additional safeguard against moisture condensing or freezing on the walls.

9.3.2 Viscometers used for silicone fluids, fluoro-carbons, and other liquids which are difficult to remove by the use of a cleaning agent, should be reserved for the exclusive use of those fluids except when calibrating. Such viscometers should be subjected to calibration checks at frequent intervals. The solvent washings from these viscometers should not be used for the cleaning of other viscometers.

10. Procedure for Transparent Liquids

10.1 In general, the viscometers used for transparent liquids are of the type listed in Table 1, A and B.

10.2 Charge the viscometer in the manner dictated by the design of the instrument, this operation being in conformity with that employed when the instrument was calibrated. If the sample contains solid particles, filter during charging through a 200 mesh (75-μm) filter.

10.2.1 With certain products that exhibit "gel-like" behavior, take care that measurements are made at sufficiently high temperatures for such materials to flow freely, so that similar results will be obtained in viscometers of different capillary diameters.

10.2.2 Allow the charged viscometer to remain in the bath long enough to reach the test temperature. Because the time varies for the different instruments and temperatures, establish a safe equilibrium time by trial (30 min should be sufficient). One bath is often used to accommodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring a flow time.

10.2.3 Where the design of the viscometer requires it, adjust the volume of the sample to the mark.

10.3 Use suction (if the sample contains no volatile constituents) or pressure to adjust the head level of the test sample to a position in the capillary arm of the instrument about 5 mm ahead of the first timing mark. With the sample flowing freely, measure, in seconds to within 0.2 s (see 6.5), the time required for the meniscus to pass from the first to the second timing mark. If this flow time is less than the specified minimum (see 9.3) select a viscometer with a capillary of smaller diameter and repeat the operation.

10.3.1 Repeat the procedure described in 10.3 to make a second measurement of flow time.

10.3.2 If two measurements agree within 0.2 %, use the average for calculating the kinematic viscosity to be reported. If the measurements do not agree, repeat the determination after thorough cleaning and drying of the viscometers and filtering of the sample.

11. Procedure for Opaque Liquids

11.1 In general, the viscometers used for opaque liquids are of the reverse-flow type listed in Table 1, C.

11.2 For steam refined cylinder oils and black lubricating oils, proceed to 11.4 ensuring a thoroughly representative sample is used. The viscosity of residual fuel oils and similar waxy products can be affected by the previous thermal history and the following procedure shall be followed to minimize this.

11.2.1 Heat in the original container, in an oven, at 60 ± 2°C for 1 h.

11.2.2 Thoroughly stir the sample with a nonmetallic rod of sufficient length to reach the bottom of the container. Continue stirring until there is no sludge or wax adhering to the rod.

11.2.3 Recap the container tightly and shake vigorously for 1 min to complete the mixing.

NOTE 3—With samples of a very waxy nature or oils of high viscosity, it may be necessary to increase the heating temperature to achieve proper mixing. The sample should be sufficiently fluid for ease of stirring and shaking.

11.3 Immediately after completing 11.2.3, pour a sufficient sample to fill two viscometers into a 100-mL glass flask and loosely stopper.

11.3.1 Immerse the flask in a bath of boiling water for 30 min.

NOTE 4: Caution—Vigorous boil-over may occur when opaque liquids that contain high levels of water are heated to high temperatures.

11.3.2 Remove the flask from the water, stopper tightly and shake for 1 min.

11.4 Charge two viscometers in the manner dictated by the design of the instrument. For example, for the cross-arm or the BS U-tube viscometers for opaque liquids, filter the sample through a 200 mesh (75-μm) filter into two viscometers previously placed in the bath. For samples subjected to heat treatment, use preheated filter to prevent the sample coagulating during the filtration. Viscometers that are charged before being inserted into the bath may need to be preheated in an oven prior to charging the sample to ensure the sample will not be cooled below test temperature.

11.4.1 After 10 min, adjust the volume of the sample to coincide with the filling marks as in the viscometer specification. For example, for the cross-arm viscometer for opaque liquids, this is described in the annex of Specification D 446. The Cannon-Fenske Opaque Viscometer design does not require further adjustment of sample volume. Allow the viscometers to reach test temperature. Because this time will vary for the different instruments and for different test temperatures, establish a safe equilibrium time by trial (30 min is normally sufficient). One bath is often used to accommodate several viscometers. Never add or withdraw a viscometer while any other viscometer is in use for measuring a flow time.

11.5 With the sample flowing freely, measure in seconds to within 0.2 s (see 6.5), the time required for the advancing ring of contact to pass from the first timing mark to the second.

11.5.1 In the case of samples requiring heat treatment described in 11.2 through 11.3.2, complete the determinations within 1 h of completing 11.3.2.

11.6 Calculate the mean kinematic viscosity ν in centistokes (millimetres squared per second) from the two determinations. For fuel oils, the two determinations should not differ by more than 1.7 % of ν at 50°C and 1.1 % of (ν + 8) at 80 and 100°C. If the two determinations exceed these limits, repeat the operation from 11.2.1. For other opaque liquids which may be non-Newtonian, the precision has not been determined.

11.6.1 Report the mean of the two determinations as the kinematic viscosity.

12. Cleaning of Viscometer

12.1 Between successive determinations, clean the viscometer thoroughly by several rinsings with an appropriate solvent completely miscible with the sample, followed by a completely volatile solvent. Dry the tube by passing a slow stream of filtered dry air through the viscometer for 2 min or until the last trace of solvent is removed.

12.2 Periodically clean the viscometer with chromic acid cleaning solution for at least twelve hours to remove residual traces of organic deposits; nonchromium-containing, strongly-oxidizing acid cleaning solutions[7] may be substituted so as to avoid disposal problems of chromium-containing solutions. Rinse thoroughly with distilled water followed by acetone, and dry with clean, dry air. Inorganic deposits may be removed by hydrochloric acid treatment before use of cleaning acid, particularly if barium salts are suspected. The use of alkaline cleaning solutions is not recommended as this can enlarge the working capillary and necessitate recalibration.

13. Procedure for Dynamic Viscosity

13.1 Determine the kinematic viscosity as described in Section 10 or 11.

13.2 Determine the density of the sample, to the nearest 0.001 g/mL at the same temperature as the viscosity, in accordance with any applicable method.

14. Calculation and Report

14.1 Calculate the kinematic viscosity, ν, from the measured flow time, t, and the instrument constant, C, by means of the following equation:

$$\nu = Ct$$

where:

ν = kinematic viscosity, cSt (mm²/s)
C = calibration constant of the viscometer, cSt/s, and
t = flow time, s.

14.2 Calculate the viscosity, η, from the calculated kinematic viscosity, ν, and the density, ρ, by means of the following equation:

$$\eta = \rho\nu$$

where:

η = dynamic viscosity, cP (mPa·s)
ρ = density, g/mL, at the same temperature used in measuring the flow time t, and
ν = kinematic viscosity, cSt (mm²/s)

14.3 Report test results for both the kinematic and dynamic viscosity rounded to the nearest one part in a thousand of the value measured or calculated, respectively.

15. Precision and Bias

15.1 The precision of this test method as obtained by statistical examination of interlaboratory test results on clean, transparent oils tested over the range from 15 to 100 cSt (59 to 212°F) using the procedure described in Section 10 is as follows:

15.1.1 *Repeatability*—The difference between successive test results, obtained by the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 0.35 % of their mean only in one case in twenty. Differences greater than this should be considered suspect.

15.1.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in normal and correct operation of this test method, exceed 0.70 % of their mean only in one case in twenty. Differences greater than this should be considered suspect.

15.1.3 The precision data[8] in 15.1.1 and 15.1.2 were obtained using five mineral oils covering the kinematic viscosity range from 3 to 1200 cSt (mm²/s) at temperatures from 38 and 99°C.

15.2 The precision[9] of this test method as obtained by statistical examination of interlaboratory test results on residual fuel oils of 30 to 1300 cSt (mm²/s) at 50°C, and oils to 170 cSt (mm²/s) at 80 and 100°C, using the procedure described in Section 11 is as follows:

15.2.1 *Repeatability*—The difference between successive test results, obtained by the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of this test method, exceed 1.5 % of their mean for results at 50°C and 1.3 % of their mean plus 8 cSt (mm²/s) [that is, 1.3 % of (mean + 8 cSt (mm²/s))] for results at 80 and 100°C only in one case in twenty. Differences greater than this should be considered suspect.

15.2.2 *Reproducibility*—The difference between two single and independent test results obtained by different operators working in different laboratories on identical test material would, in the long run, in normal and correct operation of the test method, exceed 7.4 % of their mean for results at 50°C and 4.0 % of their mean plus 8 cSt (mm²/s) [that is, 4.0 % of (mean + 8 cSt (mm²/s))] for results at 80 and 100°C only in one case in twenty. Differences greater

[7] A commercial source for a non-chromium containing cleaning solution is Godax Laboratories Inc., 480 Canal Street, New York, NY 10013.

[8] Supporting data are available from ASTM Headquarters. Request RR:D02-1132.

[9] Supporting data are available from ASTM Headquarters. Request RR:D02-1198.

than this should be considered suspect.

15.3 *Bias*—No justifiable statement can be made on the bias of the procedure in Test Method D 445 for measuring kinematic viscosity because all determinations are relative to a calibration fluid.

APPENDIX

(Nonmandatory Information)

X1. ICE POINT DETERMINATION

X1.1 To achieve an accuracy of ±0.02°C for calibrated kinematic viscosity thermometers, it is required that a check at the ice point be made. It is recommended that the interval of checking be every six months; for a new thermometer, check monthly for the first six months.

X1.2 A detailed procedure for the measurement of the ice point is described in Method E 77. The suggestions in the following sections of this appendix are given specifically for the mercury-in-glass "kinematic viscosity" thermometers described in Table 2, and may not apply to other thermometers.

X1.2.1 The ice point reading of kinematic viscosity thermometers shall be taken 5 min after being at test temperature for not less than 3 min. The ice point reading shall be expressed to the nearest 0.01°C or 0.02°F.

X1.2.2 Select clear pieces of ice, preferably made from pure water. Discard any cloudy or unsound portions. Rinse the ice with distilled water and shave or crush into small pieces, avoiding direct contact with the hands or any chemically unclean objects. Fill the Dewar vessel with the crushed ice and add sufficient distilled and preferably precooled water to form a slush, but not enough to float the ice. Insert the thermometer packing the ice gently about the stem, to a depth sufficient to cover the 0°C (32°F) graduation. As the ice melts, drain off some of the water and add more crushed ice.

X1.2.3 Raise the thermometer a few millimetres after at least 3 min have elapsed, tap the stem gently, and observe the reading. Successive readings taken at least 1 min apart should agree within one tenth of a division.

X1.2.4 Alternatively, some of the ice may be heaped around the stem above the ice point and a deep narrow channel formed to permit observation of the meniscus which is thus kept well below the general level of the ice. Observations may then be made as described above without, however, raising the thermometer.

X1.2.5 Record the readings and compare with previous readings. If the readings are found to be higher or lower than the reading corresponding to a previous calibration, readings at all other temperatures will be correspondingly increased or decreased.

STANDARD
OPERATING
PROCEDURE

Subject or Title: **Specific Gravity (Hydrometric)** Page 1 of 4

SOP No.:	Revision No.:	Effective Date:
LM-RMA-1101	**Original**	**May 1, 1989**

Supersedes: **None**

1. Scope and Application

   1.1 This method covers the determination of specific gravity by hydrometry.

   1.2 Detection Limit

   A detection limit for specific gravity is not defined.

   1.3 Applicable Matrices

   This method is applicable to the analysis of drinking, surface and saline waters that are free of oil separable by filtration.

   1.4 Dynamic Range

   The approximate dynamic range of this method is 0.7 to 2.0.

   1.5 Approximate analytical time is 10 minutes per sample.

2. Summary of Method

   2.1 A volume of filtered sample is placed in a graduated cylinder and stabilized to 15.6° C in a circulating water bath. A hydrometer is immersed in the sample and the specific gravity is read directly from the graduated scale.

Prepared by:	Kurt C. Ulll	Date: 4/28/89
Management Approval:	Julie W. Pratt II	Date: 4/28/89
QA Officer Approval:	Lisa Rodenberger	Date: 4/30/89

3. Comments

   3.1 Interferences

      3.1.1 Samples must be free of oil before measurement, therefore, only freshly filtered samples should be used.

4. Safety Issues

   4.1 All employees are expected to be familiar with and follow the procedures outlined in the Enseco/RMAL safety plan. Lab coats and safety glasses are required in all laboratory areas at all times. If you have any questions or safety concerns, see your supervisor or safety officer.

5. Sample Collection and Preservation

   5.1 Samples are to be collected in glass or plastic bottles. Any further attempts at preservation will alter the specific gravity of the sample.

   5.2 The holding time for specific gravity is not defined. Enseco recommends analysis within 28 days of collection.

6. Apparatus

   6.1 Circulating water bath, calibrated to 15.6$\pm$ 1^{o}C

   6.2 500 mL graduated cylinder

   6.3 Hydrometer with graduations no greater than 0.002

7. Reagents and Standards

   7.1 DCS Solution

      Any solution of known specific gravity can be used. Enseco recommends deionized water (ASTM D 1193, Type II) with a true value of 1.0000 $\pm$ 0.009.

8. Procedure

   8.1 Place a sufficient quantity of glass wool, to trap any separable oil, into a glass or plastic funnel.

8.2 Place the funnel into a clean, dry 500 mL graduated cylinder and gravity filter approximately 250 mL of sample. Discard glass wool when finished.

8.3 Place the graduated cylinder containing the filtered sample into a heated, circulating water bath calibrated to 15.6 ± 1°C and allow to equilibrate for one hour.

8.4 Place a hydrometer into the sample such that it does not migrate to the sides of the graduated cylinder. The hydrometer must float freely in the sample and not touch the sides of the graduated cylinder.

Read the specific gravity directly from the scale on the hydrometer.

If the range on the first hydrometer is not within the sample value, another hydrometer with the appropriate range must be used. A good "rule of thumb" would be to start with the range of 0.9-1.0 units. If the hydrometer sinks below the 0.9 units a hydrometer with the scale of 0.8-0.9 or 0.7 to 0.8 will be needed. If the hydrometer floats above the 1.0 unit mark, a hydrometer with a scale of 1.0 to 1.2 or greater will be needed.

9. QA/QC Requirements

9.1 QC Samples

9.1.1 Two DCS samples are required with every batch of 20 or less samples.

9.1.2 Duplicates may be required as project specific QC.

9.1.3 Spiking of specific gravity is not applicable.

9.2 Acceptance Criteria

9.2.1 Control limits have not been formally established. Until a substantial body of data can be obtained, control limits will be:

Accuracy	Precision
99-101%	2%

10. Reporting

10.1 Reporting units for specific gravity are not applicable.

10.2 Significant Figures

Range	Waters
< 0.999	3
1+	4

11. References

11.1 Method Source:

ASTM D 1429-76 (Reapproved 1981). "Standard Test Method for Specific Gravity of Water and Brine," Method D, pp. 231-234.

11.2 Deviations from source methods and rationale

11.2.1 Samples are analyzed ONLY at 15.6°C (60°F). Corrections from other temperatures to 60°F are not necessary.

STANDARD
OPERATING
PROCEDURE

Subject or Title: Toxicity Characteristic Leaching Procedure (TCLP)

SOP No.:	Revision No.:	Effective Date:
LM-RMA-5002	Original	September 25, 1990

Supersedes: None

1. Scope and Application

1.1 Analytes

The TCLP is designed to determine the mobility of certain organic and inorganic contaminants. The specific list of analytes may be found in Appendix I.

1.2 Reporting limits

The reporting limits are based on the individual samples as well as the individual analysis techniques. However, the sample is determined to be hazardous if it contains any analyte at levels greater than or equal to the regulatory limits. The specific list of analytes and their regulatory limits may be found in Appendix I.

1.3 Applicable Matrices

The TCLP is applicable to liquid, solid and multiphase wastes.

1.4 Dynamic Range

Consult the individual analysis SOPs (see Section 15.2).

1.5 Analysis Time

Consult the individual analysis SOPs (see Section 15.2).

Prepared by: Will Pratt	Date: September 18, 1990
Management Approval: W Pratt II	Date: 9/25/90
QA Officer Approval: Gary Troy	Date: 9/25/90

2. Method Summary

The TCLP is designed to generate an aqueous leachate of a waste sample for analysis of various organic and inorganic constituents. This leachate is intended to represent leaching the waste would experience under the condition of co-disposal with municipal waste in a sanitary landfill.

The sample is filtered to separate the solid and liquid portions. The solid portion is leached with an appropriate leaching solution. The resulting solid phase leachate is recombined with the initial liquid filtrate. The resulting TCLP leachate is then prepared and analyzed by appropriate methodology.

Two types of leachate may be generated: one for analysis of Non-volatile constituents (semi-volatile organics, pesticides, herbicides and metals) or one from a Zero Headspace Extractor (ZHE) for analysis of Volatile organic constituents.

3. Comments

Various terminology is used in this procedure which may be ambiguous. To clarify, "leachate" is used to refer to the TCLP solution generated from this procedure. This leachate may be analyzed directly for volatile constituents, prepared for non-volatile organics by an appropriate "extraction" or prepared for metals by "digestion".

3.1 Interferences

3.1.1 Due to some shortcomings of the method, losses of volatile compounds may occur. Extra care should be observed during the ZHE procedure to ensure that such losses are minimized.

3.1.2 All pH probes are to be calibrated daily (see Appendix II for the calibration procedure).

3.1.3 The leaching fluids MUST be prepared correctly. If the desired pH range is not achieved and maintained, the TCLP may yield erroneous results due to improper leaching.

3.2 Helpful Hints

3.2.1 If a "Total" analysis of the waste demonstrates that individual contaminants are absent, or present in such low concentrations that the regulatory limits could not possibly be exceeded, the TCLP need not be performed.

3.2.2 The ZHE device has approximately a 500-mL capacity. Based on the need to add an amount of extraction fluid equal to 20 times the mass of the "solid" phase, the ZHE can therefore accommodate a maximum of 25 grams of "solid" (see Section 10.7).

4. Safety Issues

4.1 Proper protective gear (i.e. safety glasses, gloves, lab coat, etc.) should be worn at all times.

4.2 Samples should be opened and handled in a properly vented fume hood.

4.3 Concentrated reagents should be handled in a properly vented fume hood. Appropriate caution should be used when handling diluted reagents.

4.4 Gas pressurized equipment is employed in this procedure. Be sure all valves and gauges are operating properly and that none of the equipment, especially tubing, is over-pressurized.

4.5 Consult the Enseco Health and Safety Manual and specific Material Safety Data Sheets (MSDS) for additional information.

5. Sample Collection, Preservation, and Holding Times

5.1 Samples being analyzed for organic compounds should be collected and stored in glass, air-tight containers to minimize the loss of volatiles. Samples being analyzed for metals only may be collected in glass or plastic containers.

5.2 Samples should be refrigerated to 4°C. No preservatives shall be added until AFTER leachate generation (see Sections 9.20 and 10.23).

5.3 Samples are subject to appropriate treatment within the following time periods (days):

From: / To:	Collection TCLP Leach	TCLP Leach Preparation	Preparation Analysis	Total Elapsed Time
Volatiles	14	N/A	14	28
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Other metals	180	N/A	180	360

6. Apparatus

6.1 Stainless steel pressure filtration apparatus (142 mm dia.), capable of 0-50 psi.

6.2 Vacuum filtration apparatus, capable of 0-50 psi.

6.3 Borosilicate glass fiber filters, 0.6 - 0.8 um (Whatman GF/F 14.2 cm, 0.7 um or equivalent).

6.4 Cellulose acetate filters, 0.65 um (Corning 25932-200 custom or equivalent), for Metals ONLY.

6.5 Zero Headspace Extractor (ZHE), gas-pressure actuated, Millipore YT30090HW or equivalent.

6.6 ZHE rotary agitation apparatus, multiple-vessel, Associated Design and Manufacturing Company 3740-6 or equivalent.

6.7 Gas-tight syringes, 100 mL capacity, Hamilton 0158330 or equivalent.

6.8 Top loading balance, 0.01 g min/4000 g max capability.

6.9 pH meter and probe, capable of reading to the nearest 0.01 unit.

6.9 pH probes and splitter box.

6.10 Magnetic stirrer/hotplate and stirring bars.

6.11 VOA vials, 20 mL, with caps and septa.

6.12 Glass jars, 1/2 - 1 gallon, with teflon lid-inserts.

6.13 Nalgene plastic bottles, 1 liter.

6.14 Miscellaneous laboratory glassware and equipment.

7. Reagents and Standards

7.1 Reagent water: For Non-volatile constituents, ASTM Type II water (Milli-Q or equivalent). For Volatile constituents, water must be passed through an activated carbon filter bed (Milli-Q or tap water passed through 500 g activated carbon).

7.2 Hydrochloric acid, 1 N: Carefully add 85 mL concentrated reagent grade HCl to 915 mL Milli-Q. Cap and shake to mix well.

7.3 Nitric acid, 1 N: Carefully add 64 mL concentrated reagent grade HNO_3 to 936 mL Milli-Q. Cap and shake to mix well.

7.4 Sodium hydroxide, 1 N: Carefully add 40 g reagent grade NaOH pellets to 1 L Milli-Q. Cap and shake until completely dissolved.

7.5 Acetic acid, glacial: Concentrated, reagent grade liquid (HOAc).

7.6 pH buffer solutions: 7, 4, 10. Commercially available.

7.7 Leaching Fluids: The pH of both solutions should be monitored daily and the pH probes are to be calibrated daily (see Appendix II).

7.7.1 Fluid #1: Carefully add 6.25 mL glacial HOAc and 64.55 mL of 1 N NaOH to 500 mL Milli-Q in a 1 gallon glass jar. Dilute to a final volume of 1 L with Milli-Q, cap and shake to mix well. When correctly prepared, the pH of this solution is 4.93 +/- 0.05.

7.7.2 Fluid #2: Carefully add 4.96 mL glacial HOAc to 500 mL Milli-Q in a one gallon glass jar. Dilute to a final volume of 1 L with Milli-Q, cap and shake to mix well. When correctly prepared, the pH of this solution is 2.88 +/- 0.05.

7.8 Nitric acid, 50% solution: Slowly and carefully add 500 mL concentrated HNO_3 to 500 mL Milli-Q. Cap and shake to mix well.

8. Procedure: Preliminary Evaluations

NOTE: Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot MAY undergo the actual TCLP extraction for Non-volatiles ONLY if it has NOT been oven dried as in Section 8.2.10. If the solid portion IS oven dried, a separate aliquot must be used for the actual leaching procedure.

8.1 Determination of Required Analyses

8.1.1 Determine from the TCLP backlog which samples require the generation of TCLP leachates and which leachates are to be generated (volatiles, etc.).

8.1.2 Consult the holding times for the appropriate tests (Section 5.3) and prioritize such that they ARE NOT EXCEEDED.

8.1.3 Determine the total volume of TCLP leachate (solid phase leachate + liquid filtrate) that needs to be generated according to the following:

Prep	Analysis	Req'd Volume
P-TCLP-VOA	Volatiles	3 x 20 mL
P-TCLP-C/EXORG	Semi-volatiles	400 mL
"	Pesticides	200 mL
"	Herbicides	200 mL
P-TCLP-C/M	Metals	150 mL

8.2 Determination of Percent Solids

8.2.1 Examine the sample and determine the type of filtration to employ. If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e. it is a solid), then proceed to Section 8.3 (Particle-size Reduction). If the sample is mostly non-viscous liquid (water or thin oil), vacuum filtration may be used. If the sample is viscous (sludge or high solids content), use pressure filtration.

8.2.2 Pre-weigh the filter device and the filtrate collection vessel. Record both masses on the benchsheet. Assemble the filtration apparatus (use blunt forceps to handle the filter membrane).

8.2.3 Homogenize the waste and weigh out a 100 g minimum subsample. Quantitatively transfer the sample to the filter apparatus and record the mass of any remaining residue. Calculate and record the mass of sample.

8.2.4 Slowly apply gentle pressure or vacuum of 10 psi. Allow the sample to filter until no SIGNIFICANT additional liquid has passed through the filter in a 2 minute period.

8.2.5 Repeat previous step up to 50 psi max in 10 psi increments. Remove the filtrate collection vessel and determine and record its mass.

8.2.6 Determine and record the mass of the initial filtrate. Retain the filtrate for use in Section 8.5.

8.2.7 Determine and record the "solid" phase mass by subtracting the mass of the initial filtrate from the mass of the subsample. Retain the solid phase for use in Section 8.3.

NOTE: Some samples will contain liquid material that does not filter (e.g. oil). Do not attempt to filter the sample again. The "solid" phase is defined as any material remaining in the filtration apparatus.

8.2.8 Determine and record the % Solids (see Section 12.1):

If the % Solids is slightly greater than or equal to 0.5% and it is believed that some of the mass is due to moisture entrained in the filter, proceed to 8.2.9.

If the % Solids is significantly $>$ 0.5 %, proceed to Section 8.3.

If the % Solids is $<$ 0.5 %, discard the solid phase. No leaching will be necessary; the filtrate will be considered the TCLP leachate. Proceed to Section 8.5.4.

8.2.9 Remove the solid phase and filter from the filtration apparatus.

8.2.10 Dry the filter and solid phase at 100 +/- 20°C until two successive weighings yield the same value within +/- 1%. Record the final weight. Return to 8.2.8.

8.3 Particle-size Reduction

Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e. capable of passing through a 9.5 mm (0.375 inch) standard sieve). Record on the benchsheet.

NOTE: Consult your supervisor or manager when dealing with unusual sample matrices (e.g. wood, cloth, metal, brick).

8.4 Determination of Appropriate Leaching Fluid

8.4.1 Weigh out a 5.0 g sub-sample of the solid phase into a 250-mL beaker.

8.4.2 Add 96.5 mL of Milli-Q water, cover with a watchglass, and stir for 5 minutes on a hotplate/stirrer.

8.4.3 Measure and record the pH. If the pH is LESS THAN OR EQUAL to 5.0, use Fluid #1 and proceed to Section 8.5.

8.4.4 If the pH is GREATER than 5.0, add 3.5 mL 1 N HCl, cover with a watchglass and while stirring, heat at 50°C for 10 minutes. Remove, cool.

8.4.5 Again, measure and record the pH. If the pH is less than or equal to 5.0, use Fluid #1. If the pH is greater than 5.0, use Fluid #2.

8.5 Determination of Filtrate/Leachate Compatibility

8.5.1 Place 5 mLs of the appropriate leaching fluid (determined in the previous step) into a blood dilution vial.

8.5.2 Add 5 mLs of the initial filtrate, cap and shake. Examine for miscibility and record on the benchsheet.

8.5.3 If the phases are miscible, the initial filtrate and solid phase leachate may be physically recombined upon completion of the leachate generation. If the phases are NOT miscible, the initial filtrate and the solid phase leachate should be prepared and analyzed separately and the results mathematically combined (see Section 12.2).

8.5.4 For samples requiring TCLP Semi-volatiles, Pesticides, Herbicides or Metals analysis, proceed to Section 9.

8.5.5 For samples requiring TCLP Volatiles (ZHE) analysis, proceed to Section 10.

9. Procedure: Non-volatile Constituents: Semi-volatiles, Pesticides, Herbicides, Metals (Bottle leach)

NOTE: The aliquot used in the Preliminary Evaluation MAY be used for this procedure ONLY if it was not oven dried.

NOTE: All masses should be recorded to the nearest 0.01 g.

9.1 Examine the sample and determine the type of filtration to employ. If the waste will obviously yield no free liquid when subjected to pressure filtration (i.e. it is a solid), then homogenize the sample, weigh out an appropriate size subsample, record the mass, and proceed to Section 9.10. If the sample is mostly non-viscous liquid (water or thin oil), vacuum filtration may be used. If the sample is viscous (sludge or high solids content), use pressure filtration.

9.2 Pre-weigh the filter device and the filtrate collection vessel. Record both masses on the benchsheet. Assemble the filtration apparatus (use blunt forceps to handle the filter membrane). If evaluating for the mobility of metals, wash the filter with dilute HNO_3 and rinse with Milli-Q.

9.3 Determine the optimum sample size to use based on the required analyses (Section 8.1) and the preliminary percent solids data (Section 8.2). A sufficient volume of final leachate should be generated to support all required analyses.

9.4 Prepare one leachate blank PER 20 SAMPLES, PER LEACHATE FLUID, PER LEACH VESSEL TYPE (this yields 3 possibilities: ZHE, Bottle-Fluid #1, Bottle-Fluid #2). This allows for samples to be leached on different days without leaching a blank every day. Samples which are NOT leached (merely filtered) will ALSO be associated with these blanks.

9.5 Homogenize the waste. Weigh out the subsample into the filtration device and record the mass. Quantitatively transfer the sample to the filter apparatus and record the mass of any remaining residue.

9.6 Slowly apply gentle pressure or vacuum of 10 psi. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a 2 minute period.

9.7 Repeat previous step up to 50 psi max in 10 psi increments. Remove the filtrate collection vessel and determine and record its mass.

9.8 Determine and record the volume (mass) of the initial filtrate. Cover with aluminum foil and retain for use in Section 9.17.

9.9 Determine and record the "solid" phase mass by subtracting the mass of the liquid filtrate from the mass of the subsample.

9.10 Evaluate the solid portion of the waste for particle size. If it contains particles greater than 1 cm in size, prepare the solid portion of the waste for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e. capable of passing through a 9.5 mm (0.375 inch) standard sieve). Record on the benchsheet.

NOTE: Consult your supervisor or manager when dealing with unusual sample matrices (e.g. wood, cloth, metal, brick).

9.11 Determine the minimum total volume of solid phase leachate that needs to be generated. Refer to Section 8.1.3.

9.12 Divide the total volume of solid phase leachate required by 20 to determine the mass of solid phase required for leaching. Round this mass UP to the nearest 5 g.

9.13 Weigh the required mass of solid phase into an appropriate bottle (plastic for Metals only, glass for all others) and slowly add 20 times its mass of appropriate leaching fluid (Section 8.4). (For example, 20 g of sample would require 400 mL of leaching fluid).

9.14 Cap the bottle tightly, secure in a rotary agitator and rotate end-over-end at 28-32 rpm for 16-20 hours.

NOTE: The bottle may be removed and opened periodically in a properly vented hood to relieve any built-up pressure.

9.15 Remove the bottle and filter the sample using vacuum or pressure filtration. The entire sample need not be filtered, however, sufficient volume should be generated to support the required analyses. See Section 8.1.3. Cellulose filters may be used for leachates being prepared for metals only.

9.16 If the waste contained no initial filtrate, this solution is defined as the TCLP leachate.

9.17 If the waste DID yield an initial filtrate, consult the benchsheet for initial filtrate/leachate compatibility. If they are compatible, they are to be combined in the correct proportions (see Section 12.2) and mixed well. This combined solution is defined as the TCLP leachate.

9.18 If the individual phases are NOT compatible, they are to be prepared and analyzed separately and the results combined mathematically. See Section 12.3 and the attached example.

9.19 Measure and record the pH of the TCLP leachate. (Do not attempt to measure the pH of oily samples as the probe may be rendered inoperable.)

9.20 Immediately preserve the leachate as follows:

Metals	pH < 2 w/50% HNO_3 for non-oils.
Others	Refrigerate to $4^{o}C$.

9.21 Label each sample with the appropriate information and submit to the appropriate analytical groups for prep and analysis. Also submit copies of the prep benchsheets with the samples. See Sections 8.1 and 15.2 for additional information.

10. Procedure: Volatile Constituents (ZHE)

NOTE: Use the ZHE device to obtain a TCLP leachate for analysis of volatile compounds only. Leachate resulting from the use of the ZHE shall NOT be used to evaluate the mobility of non-volatile analytes (e.g. metals, pesticides, etc.).

NOTE: All masses should be recorded to the nearest 0.01 g.

10.1 Assemble the ZHE apparatus, close all valves except the gas inlet/outlet valve and pressurize to 50 psi. Allow to stand for 1 hour and check the pressure on the built-in gauge to make sure it is not leaking. If the pressure is NOT 50 psi, consult your supervisor. Disassemble the ZHE leaving the piston in the ZHE body.

10.2 Adjust the ZHE piston in the ZHE body to the appropriate height (slightly moisten the O-rings with leaching fluid if necessary).

10.3 Consult the benchsheet and examine the sample. If the sample appears to be different from the preliminary information found on the benchsheet, consult your supervisor. If the sample is < 100% solids (i.e. yielded free liquid upon filtration), proceed to Section 10.6.

10.4 Prepare one leachate blank PER 20 SAMPLES. This information will need to be tracked for the purpose of assigning Prep QC Lot numbers. This allows for samples to be leached on different days without leaching a blank every day.

10.5 Homogenize the waste. If the preliminary evaluations indicated the need for particle size reduction, weigh out a sufficient size subsample and prepare for leaching by crushing, cutting, or grinding such that all particles are less than 1 cm in size (i.e. capable of passing through a 9.5 mm (0.375 inch) standard sieve; do NOT sieve the sample, however). Record on the benchsheet.

NOTE: Consult your supervisor or manager when dealing with unusual sample matrices (e.g. wood, cloth, metal, brick).

10.6 Place the ZHE apparatus on the balance and tare the balance.

10.7 Determine the appropriate size subsample to weigh using the percent solids information from Section 8.2. For wastes containing < 5.0 % solids, weigh out enough sample to support the volatiles analyses (Section 8.1) since the the liquid portion of the waste after filtration is defined as the TCLP leachate.

10.8 Homogenize and weigh an appropriate size subsample of the waste into the ZHE and record the mass. Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

10.9 Place the ZHE in a vertical position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

10.10 Carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure. If the waste is 100% solid, slowly increase the pressure to a maximum of 50 psi to force out as much headspace as possible and proceed to Section 10.14.

10.11 Assemble a syringe and place the plunger in all the way. Adjust the tension on the plunger to provide slight drag. Attach the syringe to the liquid inlet/outlet valve and open the valve.

10.12 Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a 2 minute period. If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe and return to Section 10.11.

10.13 Repeat previous step up to 50 psi max in 10 psi increments. Remove the syringe and record the total filtrate volume. Close the valve and discontinue gas pressure. Transfer the filtrate to VOA vials and label appropriately.

NOTE: If the original waste contained < 5.0 % solids (Section 8.2), this filtrate is defined as the TCLP leachate and you may proceed to Section 10.24. Otherwise, save the vials for recombination as in Section 10.22.

NOTE: The material remaining in the ZHE is defined to be the "solid" phase.

10.14 Based on the information from Sections 8.2 and 10.7, determine the volume of Fluid #1 to load into the ZHE on the "solid" phase.

NOTE: The ZHE prep uses ONLY Fluid #1.

10.15 Load the fluid transfer reservoir with an excess of Fluid #1 and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.

10.16 Attach the transfer line to the liquid inlet/outlet valve and open the valve. Carefully pump the required volume into the ZHE and close the valve. Disconnect the transfer line.

10.17 Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) 2 or 3 times. Reposition the ZHE in the vertical position.

10.18 Pressurize the ZHE to 5-10 psi. Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.

10.19 Repressurize the ZHE to 5-10 psi and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours.

10.20 Confirm that the pressure of 5-10 psi was maintained throughout the leaching. If it was NOT maintained, return to Section 10.1 and repeat the leachate with a new aliquot of sample.

10.21 Attach a syringe and open the liquid inlet/outlet valve to collect the aqueous leachate.

10.22 If the waste contained an initial filtrate (Section 10.12) that is miscible with the solid phase leachate (as determined in Section 8.5), the solid phase leachate may be directly recombined in the correct proportions (see Section 12.2) with the initial filtrate. If the individual phases are NOT compatible, they are to be collected, prepped and analyzed separately.

10.23 Following collection, store the TCLP leachate in 3 20-mL VOA vials with minimal headspace at 4°C and prepare for analysis as soon as possible using the appropriate organic extraction procedure (see Section 15.2).

10.24 If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.3.

11. QA/QC Requirements

11.1 QC Samples

11.1.1 One blank per Fluid type per extraction vessel type (using the same leaching Fluid as used for the samples) shall be prepared for every twenty samples (whether leached or not). The blanks are generated in the same way as the samples.

11.1.2 Duplicate Control Samples (DCSs) are required with each batch of 20 or fewer samples. The DCSs shall be generated AFTER the TCLP leachate at the time of the preparative digestion or extraction. Consult the individual analysis SOPs (see Section 15.2).

11.1.3 Duplicates may be required for project specific QC.

11.1.4 Matrix spikes may be required for project specific QC. They shall be added AFTER the TCLP leachate is generated but BEFORE PRESERVATION.

11.2 Acceptance Criteria

11.2.1 Consult the Enseco QAPP and Internal QC SOP (M-EQA-002) for method blank acceptance criteria.

11.2.2 Consult the individual analysis SOPs for DCS recovery criteria.

11.2.3 Acceptance criteria for project specific duplicates have not been established.

11.2.4 Acceptance criteria for project specific matrix spikes have not been established.

11.3 Corrective Actions

11.3.1 Consult the Enseco QAPP and Internal QC SOP (M-EQA-002) for corrective action for blanks and DCSs.

11.3.2 No corrective actions have been defined for duplicates.

11.3.3 Evaluation of matrix spike results and recovery correction of data based on same will not be employed by Enseco but will be the sole responsibility of the client. See Section 15.3.5.

11.3.4 If the ZHE exhibits loss of pressure, repressurize to 50 psi and submerge in water to check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, replace O-rings as necessary. Retest the device. If leakage problems cannot be resolved, the ZHE is unsuitable for use and the manufacturer should be contacted.

12. Calculations

12.1 Calculation of % Solids:

$$\frac{\text{Mass, "solid" phase}}{\text{Mass, initial subsample}} * 100$$

12.2 Calculation of volume of initial filtrate phase to recombine with solid phase extract:

$$\frac{\text{Mass, extracted solids}}{\text{Mass, solids}} * \text{Total Volume, initial filtrate}$$

12.3 Mathematical recombination of analytical results:

$$\text{Final Analyte Concentration} = \frac{(V_1 * C_1) + (V_2 * C_2)}{V_1 + V_2}$$

where V_1 = total volume of the initial filtrate phase (L),
C_1 = analyte concentration in initial filtrate phase (mg/L),
V_2 = volume of the theoretical solid phase extract (L),
C_2 = analyte concentration in solid phase extract (mg/L),

and where V_2 = total solid phase mass * 20.

13. Reporting Requirements

13.1 Units

Regardless of the nature of the sample, all results are reported in units of mg/L.

13.2 Limits, Significant Figures

Consult the appropriate analytical methods (Section 15.2).

13.3 LIMS Data Entry

13.3.1 Enter the QC Lot number of the leachate blank in the TCLP leachate prep test.

13.3.2 The usual standards for the remaining data entry apply.

13.4 Anomalies

All anomalies observed during the leach procedure must be noted on an anomaly form. Some examples of such anomalies are:

13.4.1 Sample was monolithic - subsample was obtained by (crushing, cutting, grinding, sawing, etc.).

13.4.2 Insufficient sample - less than the required 100 g minimum was available.

13.4.3 Multiple phases - "X" phases were present.

13.4.4 Sample was oil - single phase.

13.4.5 Sample contained liquid which did not filter under test conditions.

13.5 Data Package

The original TCLP data package will contain the following information: Cover sheet, Special Instructions (only when applicable), Leachate benchsheet, Blank tracking form and any additional information. This will be filed normally and a copy of the data package will be submitted to each analytical group (as appropriate) with each batch of leachates generated.

14. Review Requirements

14.1 Review all applicable holding times. If a holding time was exceeded, confirm that a holding time violation form was properly documented and routed.

14.2 If Total analysis results are available, those results may be compared with the TCLP analysis results according to the following:

$$\text{Total} >= \text{TCLP} * \frac{\{(\text{Solids mass (g)} * 20) + \text{Filtrate volume}\}}{1000 * \text{sample mass used (kg)}}$$

NOTE: When the sample is 100% Solids, this equation reduces to:

$$\text{Total} >= \text{TCLP} * 20$$

15. References

15.1 Method Source: Toxicity Characteristic: Corrections to Final Rule. Method 1311, Federal Register, Vol. 55, No. 126, Friday, June 29, 1990.

15.2 Related Documents

15.2.1 Toxicity Characteristic: Final Rule. Method 1311, Federal Register, Vol. 55, No. 61, Thursday, March 29, 1990.

15.2.2 Technical Background Document and Response To Comments, Method 1311, Toxicity Characteristic Leaching Procedure, USEPA/OSW, April, 1989.

15.2.3 LM-RMA-2002: [TCLP] Metals: Acid Digestion

15.2.4 LM-RMA-2005: Mercury

15.2.5 LM-RMA-2006: GFAA (Se)

15.2.6 LM-RMA-2001: ICP

15.2.7 LM-RMA-3001: VOAs

15.2.8 LM-RMA-3013: BNAs

15.2.9 LM-RMA-4003: Pesticides

15.2.10 LM-RMA-4007: Herbicides

15.3 Deviations from Source Method and Rationale

15.3.1 Section 8: Preliminary Evaluations. Section 7.1 of the source method states that the sample aliquot used for the preliminary evaluation "...may not actually undergo TCLP extraction". No reason is given there or in the background document. It is assumed that the reasoning is that solid phase degradation may occur upon heating to determine % dry solids. In order to reduce the time involved, and based on said assumption, the sample used for the preliminary evaluation may be used for the actual TCLP leach PROVIDED THE SAMPLE WAS NOT OVEN DRIED during the % solids determination.

15.3.2 Section 8.2.4: Preliminary Determination of Percent Solids. The wording of Section 7.1.1.7 of the source method has been modified. Filtration proceeds until "...no SIGNIFICANT additional liquid has passed through the filter..." to allow for judgement in determining the total time required for filtration.

15.3.3 Section 8.5: Preliminary Determination of Filtrate/Leachate Compatibility. Section 7.2.13 of the source method provides no guidance as to how to make this determination. As a result, the procedure herein was developed and incorporated into the Preliminary Determinations section.

15.3.4 Section 9.4: Leachate Blanks. Section 8.1 of the source method states that a minimum of one blank for every 20 extractions "...that have been conducted in an extraction vessel." Enseco-RMAL has interpreted this to mean one blank per twenty samples leached per TYPE of leaching vessel (i.e. Bottle or ZHE) per leach fluid used.

15.3.5 Section 9.15: Filtration. Section 7.2.12 of the source method requires the use of glass fiber filters for the filtration of the Non-volatiles TCLP leachates. Cellulose filter material has been deemed acceptable by Enseco for the filtration of metals only.

15.3.6 Section 10.21: Volatiles Filtrate Collection. Section 7.3.9 of the source method requires that the mass of ZHE filtrate be determined. Enseco-RMAL currently uses graduated syringes for collection and therefore determines ZHE filtrate volume.

15.3.7 Section 11.1.4: QA/QC - Matrix Spikes. Section 8.2 of the source method states "A matrix spike shall be performed for each waste type..." and "A minimum of one matrix spike must be analyzed for each analytical batch." Because of the diverse nature of samples that Enseco-RMAL receives, the logical conclusion of this requirement would be to spike every sample. Further, Section 8.2.3 of the source method states "The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist." The standard Enseco QAPP is designed to address the performance monitoring of analytical methodology through the LCS program. As a result, Enseco-RMAL will perform matrix spikes only as requested by the client and will NOT perform calculations for recovery OR correct for alleged analytical bias. The use and interpretation of the matrix spike results shall be the sole responsibility of the client.

APPENDIX I

Toxicity Characteristic Analytes and Regulatory Levels

(Final Rule)

Contaminant	mg/L
Arsenic	5.0
Barium	100.0
Benzene	0.5
Cadmium	1.0
Carbon tetrachloride	0.5
Chlordane	0.03
Chlorobenzene	100.0
Chloroform	6.0
Chromium	5.0
Cresols	200.0
2,4-D	10.0
1,4-Dichlorobenzene	7.5
1,2-Dichloroethane	0.5
2,4-Dinitrotoluene	0.13
1,1-Dichloroethylene	0.7
Endrin	0.02
Heptachlor	0.008
Hexachlorobenzene	0.13
Hexachlorobutadiene	0.5
Hexachloroethane	3.0
Lead	5.0
Lindane	0.4
Mercury	0.2
Methoxychlor	10.0
Methyl ethyl ketone	200.0
Nitrobenzene	2.0
Pentachlorophenol	100.0
Pyridine	5.0
Selenium	1.0
Silver	5.0
Tetrachloroethylene	0.7
Toxaphene	0.5
Trichloroethylene	0.5
2,4,5-Trichlorophenol	400.0
2,4,6-Trichlorophenol	2.0
2,4,5-TP (Silvex)	1.0
Vinyl chloride	0.2

APPENDIX II

pH Meter and Probe Calibration Procedure

Corning 255 Ion meter

1) Depress the MODE button until the meter is in the pH mode.

2) Depress the CAL key.

3) Depress the Right arrow (2 pt calibration).

4) Rinse the electrode with DI water, then with the 7 buffer.

5) Place the electrode in the 7 buffer. Depress the READ button.

6) Allow the reading to stabilize. Depress the READ button again.

7) Rinse the electrode with DI water, then with the 4 buffer.

8) Place the electrode in the 4 buffer. Depress the READ button.

9) Allow the reading to stabilize. Depress the READ button again.

10) The screen will read SAMPLE. Rinse the electrode with DI water and place the electrode in the 10 buffer. Allow the reading to stabilize. It should read between 9.5 and 10.5.

11) Rinse the electrode with DI water and store in either DI water or the 7 buffer. The meter is now calibrated and ready for use.

NOTE: If the 10 buffer does NOT read between 9.5 and 10.5, recalibrate as above. If the second attempt is unsuccessful, consult your supervisor.

NOTE: DO NOT allow the electrode to dry out. DO NOT immerse the electrode in oily samples. Consult your supervisor.

Effective Date: 7-2-91

ATOMIC ABSORPTION SPECTROMETRY
Furnace - Direct Injection

Scope and Application: Metals in solution can be readily analyzed by Atomic Absorption Spectrometry using either flame, furnace or hydride techniques. The furnace - direct injection technique allows for lower detection limits. The use of the graphite platform in furnace analyzed can improve sensitivity and reduce some matrix interferences.

Method: Furnace; direct injection

Reference: EPA 1984, Section 200
Analytical Methods for Zeeman Graphite Tube Atomizers - Varian 1986
Spectra AA - 300/400 Zeeman Operation Manual - Varian March 1988

Sample Handling: Acidify with concentrated nitric acid to ph < 2. Drinking waters and filtered groundwater samples free of particulate matters and organics may be analyzed directly, while wastewaters, leachates, solids, etc., must be digested prior to analysis (refer to appropriate digestion procedures). Samples must be analyzed within 6 months.

Reagents and Apparatus:

1. Zeeman Automatic Absorption Spectrometer - 400
2. Zeeman Graphite tube Atomizer
3. IBM Personal System/2 Model 30 Computer
4. EPSON EX-800 Printer, Citizen HSP-500 printer, or similar adaptable printer.
5. Required metal lamp and power source
6. Stock and standard solutions for required metal
7. Class A volumetric glassware
8. Instr-analyzed nitric acid
9. Deionized (D.I.) water
10. Argon gas - prepurified grade
11. Graphite partition tubes
12. Graphite plateau tubes and platforms
13. Disposable 2 mL sample cups
14. Eppendorf 100-1000 microliter pipetor
15. Disposable 10 ml beakers

Procedure:

Power Up Procedure

1. Turn on argon gas and cooling water.

2. **Always turn the system on in the following order:** spectrometer, furnace, printer, and computer. This initializes the communication relays correctly so that all components of the system can "talk" to each other.

3. After the DOS prompt has been displayed, type "Zeeman" and press Enter. After a brief pause, an introductory message will then be displayed followed by the PROGRAM MODES page. Follow the on-screen instructions to select the appropriate mode.

Automatic Run Using the Sampler:

Notes:

a. Only programs which have been stored can be used for an automatic run.

b. For all programs, the method of sample introduction (**instrument parameters** page) must be specified as **sampler automixing** (for automatic mixing of calibration standards from a single, high concentration standard) or **sampler premixed** (for a full set of calibration standards that are prepared by the operator prior to analysis).

c. Options on the **report format** page allow raw data to be printed either as it is collected during an analytical run (used for most analyses), or after the analysis is completed (used for sequential runs of multiple elements).

d. If an automatic run is stopped and then restarted, the sampler will automatically perform a tube clean and analyze a blank. It will then continue on according to the instructions set in the **sequence control** page.

e. F9 through F12 are hard keys with their function on the supplied overlay. F1 through F6 are soft keys; their functions will change from one page to the next. The function for each soft key is displayed at the bottom of the screen and only those displayed are active for that page.

f. Any page described below can be recalled by returning to the **index** and entering the appropriate page number.

1. Perform daily maintenance. Check the condition of the graphite tube and replace as necessary.

2. From the **program modes** page, press **automatic run.** The system will automatically display the **sequence selection** page.

3. On the **sequence selection** page, press F1 to clear the sequence of previous element(s) and enter the code of the program to be run. If more than one program is to be run, press **enter** after each element program number. Press F6 to recall program. The **sequence control** page will automatically be displayed.

4. Follow on-screen instructions to enter the number of initial tube burns for cleaning (1 or 2 for previously used furnace tubes, 3 or 4 burns for new tubes), the starting position for the run (usually position #1), and the last position for the run. Note that when the analysis at the last position is completed, the automatic run is terminated and the element lamp is shut off automatically. Setting the final position to leave several empty cups at the end of the analytical run allows necessary repeats or

dilutions to be added to the end of the current run, saving lamp warm-up and calibration time.

5. Return to the **index** and select page 6 **(optimization)**

   a. Open the lamp turret cover and ensure that the required lamp is in the operating position.

   b. Observe the signal bar labelled **align hc lamp** displayed on the video screen. Turn the horizontal lamp base adjusting screw (the top one of the two) fully clockwise. Now turn this screw slowly counter-clockwise until the first peak is detected (the length of the signal bar will increase). Continue adjusting this screw until the length of the signal bar is the maximum obtainable (if the signal bar is fully extended, press the rescale soft key, F1, to bring the signal bar back on scale and again adjust the screw to obtain maximum signal. Note particularly that turning the horizontal adjusting screw further counter-clockwise may produce a second peak. **Do not** align the lamp on this second peak - **always** align the lamp on the first peak. Carefully adjust the vertical adjusting screw (the bottom one of the two) so that the length of the signal bar is the maximum obtainable (if necessary, press F1 to rescale the signal bar).

   c. Record the photomultiplier voltage in the instrument log book. A constantly increasing voltage over time is evidence of decreasing efficiency of the element lamp. Monitor this voltage to determine when element lamps should be replaced.

   d. When switching from partition to platform tubes (or vice-versa), check the position of the graphite tube automizer:

      Hold a piece of white card between the righthand end of the graphite tube automizer and the sample compartment window. Use the furnace vertical adjust and position the automizer until light from the hollow cathode lamp is obviously passing through the graphite tube on to the card.

      Remove the card. Observe the signal bar labelled **align hc lamp** displayed on the video screen. Use the furnace vertical adjust and carefully adjust the position of the graphite tube automizer until the length of the signal bar is the maximum obtainable.

6. Use the soft key indicated, or return to **index** to select **standards page.** This page tells which standards are to be used for calibration.

7. Use the soft key indicated, or return to **index** to select **sampler page.** This page lists the volume of standards, blanks, samples and modifier that are to be used for analysis.

8. At the **sampler** page, press F2 to align the sampler arm. Place a finger on the arm as it starts to descend into the furnace and gently lower the arm by hand. Carefully adjust the sampler position using the two adjustment knobs on the base of the autosampler so that the capillary is exactly in the center of the sample injection hole. With the capillary down in the furnace, and using the mirror, turn the height adjusting screw so the capillary is about 1 mm above the bottom of the tube or platform.

9. Return to **index** and select **report format** page. Enter operator initials, analysis date, and batch number. Review the defaults set for the remaining parameters. Follow the on-screen instructions for using the **home** key to make any needed changes.

10. If sample labels are to be printed with the raw data, press F6 and enter appropriate labels. Note that the **Tab** key will jump the cursor to the next field; the ↑ and ↓ keys to move the cursor up and down the columns.

11. Press F10 to zero the instrument before beginning analysis. Press F11 to start the automatic run.

12. To change basic operating conditions (these are default conditions recalled automatically with the analytical program), press F12 to pause run, return to the **index** and select page 4, **instrument parameters.** Parameters may be changed using the **home** key or soft keys as indicated. Press F11 to resume the analytical run.

Furnace Maintenance:

The following maintenance is to be done each day the furnace is operated:

1. Clean the furnace windows.

    a. Twist out furnace windows from furnace unit.
    b. Wipe windows with a Q-tip moistened with alcohol.
    c. Rinse windows with D.I. water and dry with a Kim-Wipe
    d. Re-insert windows in furnace.

2. Check machine windows and clean if needed.

3. Wipe inside of furnace with a Q-tip moistened with alcohol.

4. Fill the autosampler rinse bottle with D.I. water.

5. Open the syringe compartment door on the autosampler and pull the syringe assembly carefully out of its mounting. Remove the plunger from the syringe, and on the **sampler** page, press F3 to rinse the syringe and bleed any air bubbles from the syringe. Press F3 and rinse again, while water is dripping from syringe insert the plunger into the syringe. Wipe the syringe dry and carefully re-insert in its mounting.

6. **Inserting graphite tube**

    a. Swing toggle level on top of furnace fully clockwise to open furnace.

    b. Place graphite tube in the graphite shroud in the center block. Align sample introduction part of the graphite tube with the opening in the furnace block.

    c. Swing the toggle lever fully counter-clockwise and the righthand electrode assembly will automatically close on the center block.

d. Before using a new graphite tube for analyses, use the tube clean utility (**signal graphics** page) 3-4 times to remove any contamination. This can be done automatically by entering 3 or 4 tube cleans in the appropriate field on the **sequence control** (page 11) before starting an analytical run.

Effective 7-2-91

ATOMIC ABSORPTION SPECTROMETRY FLAME-DIRECT ASPIRATION

Scope and Application: Metals in solution can be readily analyzed by Atomic Absorption Spectrometry using either flame or furnace techniques. The flame-direct aspiration can be used for most metals but is generally not as sensitive as the furnace method. Both the air-acetylene and nitrous oxide-acetylene flame techniques are described in this operating procedure as well as the use of emission spectroscopy.

Method: Flame; direct aspiration

Reference: EPA 1984, Section 200

"Analytical Methods for Flame Spectrophotometry", Varian, 1979

Spectr AA - 10/20 Operation Manual, Varian

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Sample Handling: Acidify with concentrated nitric acid to pH < 2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Samples must be analyzed within 6 months from sampling date.

Reagents and Apparatus:

1. Varian Spectr AA-20
2. Stock and standard metal solutions
3. class A volumetric glassware
4. Instra-analyzed nitric acid
5. Deionized (D.I.) water
6. Hollow cathode element lamps
7. Disposable 10 mL beakers
8. Eppendorf 100-1000 uL pipetter
9. Oxford 5 or 10 ml pipetter
10. Acetylene gas
11. Air supply
12. Nitrous oxide gas
13. Air-acetylene burner head or nitrous oxide-acetylene burner head

Setup:

1. Power on instrument. The computer will automatically start with a memory check. When the first screen appears, it is ready to operate.

Note: Allow instrument a 1/2 hour warm up period for electronic and optical components to achieve thermal equilibrium before beginning analysis.

2. Power on printer. Check the paper supply.

3. Install the desired element lamp in the lamp turret by depressing the middle white button behind the socket, inserting the lamp, and releasing the button. Ensure that the lamp is secure and that the connections are fitting properly.

   Note: Allow lamp a 10-15 minute warm up period before beginning analysis.

Procedure:

This procedure will outline an analysis as it would be run following the instructions given on sequential computer screens. **Note:** Any time during setup the "Index" key can be used to go to any screen in the software.

1. Soft key selections allow the operator to develop program, modify program, or automatic run. The typical analysis will be run by selecting "Automatic Run."

   Note: After completing required information on the present screen use the soft keys to call up the next screen.

2. **"Sequence Selection".** This screen lists the programs on file. Use the "Clear Sequence" soft key to erase the last sequence used, type in the number corresponding to the program desired, and press "Sequence Selection" soft key. This will automatically recall the program.

3. **"Sequence Control".** The screen is used for autosampler control only. Go to next screen by pressing "Report Format" soft key.

4. Use cursor arrows and numeric keys to enter operator and date. The "Home" key is used to change entries of other parameters.

5. **"Sample Labels".** Use the cursor arrows and numeric keys to enter labels.

   Note: Sample labels will only be printed if the automatic run is used.

6. **"Optimization".** This screen is used to optimize wavelength and lamp position.

   a. Ensure lamp is located correctly and is on (lamp is automatically turned on when program is called up).

   b. Select proper slit width.

c. Release brake ("off") and set approximate wavelength. Set brake ("on") and fine-tune the wavelength to achieve maximum intensity on HCl bar graph. "Rescale" (soft key) as often as necessary to keep graph on scale.

d. Optimize lamp position by adjusting the adjusting screws on back of the lamp socket. Adjust for maximum intensity on the bar graph. "Rescale" as often as necessary.

e. If background is used, adjust maximum intensity on background bar graph by 2 set screws on the background corrector housing. Set attenuator ("In" or "Out") if necessary. "Rescale" if necessary.

f. Record the photomultiplier voltage in the instrument log book. A constantly increasing voltage over time is evidence of decreasing efficiency of the element lamp. Monitor this voltage to determine when element lamps should be replaced.

Note: HCl and background lamp intensities should match as closely as possible. The attenuator will cut down background intensity. A lower lamp current will lower its intensity.

7. **Flame Ignition**

a. Turn on compressed air to 50 psi (35-65 psi)

b. Turn on acetylene tank, pressure should be 7-15 psi.

c. Turn on nitrous oxide tank (if necessary - the proper burner head must be in place for ignition to occur). Tank pressure should be 50 psi (35-65 psi).

d. Press "Ignite" key and hold down until flame ignites.

Note: Let burner head warm to equilibrium before analysis; 5 to 10 minutes for an air-acetylene flame, 10 to 15 minutes for a nitrous oxide-acetylene flame.

8. **Signal Optimization**

a. Press "Signal Optimization" soft key on optimization screen.

b. Adjust burner head using 2 adjusting screws and rotation lever for maximum intensity while aspirating a high standard.

c. Adjust the nebulizer/glass bead by slowly turning the screw directly below the nebulizer.

9. **Flame Emission Procedures**

   a. In this method, no element lamp or background correction is used. Burner head position and wavelength are optimized while aspirating the highest working standard.

   b. Turn the burner head full right or left (approximately 30° angle).

   c. Select optimization screen.

   d. Adjust wavelength for maximum intensity.

   e. Press "Emission Setup" soft key.

   f. Continue with automatic/non-auto run.

10. **Automatic Run (no autosampler)**

   Note: Only pre-existing programs can be used.

   a. Press "Start" key to initialize run. Once a run is started, it can be paused by pressing the "Stop" key, but none of the program parameters can be changed.

   b. Press "Instrument Zero" key after program has been recalled to establish a zero instrument baseline.

   c. Aspirate standards or sample and press "Read". The instrument will display the std #/sample # on the top of the screen, along with the absorbance.

   d. The "Previous Sample"/"Next Sample" soft keys can be used to repeat a specific analysis or move ahead in the sample order, "solution type" can be used to restandardize by starting at "blank".

   e. If more than 66 samples and standards are to be run, add them at the end of the run and depress "Previous Sample" key for each sample. Since the sample labels cannot be changed, leave the last few labels blank on the sample labels page, and write them in when the run is completed.

   f. Press "Stop" key to pause or end the analysis.

11. **Non-automatic Run**

   Note: A modified or newly developed program can be run in this mode, a well as a pre-existing program.

   a. Set up instrument according to previous instruction. Note that the sample labels and report format cannot be printed in this mode.

b. Advance to "Standards" screen by use of soft key on optimization screen or through the "index".

c. Aspirate standards/samples and press "Read" key as in the automatic run.

d. This mode is not limited to the samples. As no labels are printed, these must be written onto the printout by hand.

12. **Instrument Shut Down**

a. Turn off flame ("Flame Off" key).

b. turn off all gases.

c. Recall program #10 or # (Emission programs), so that no lamp is turned on unnecessarily when the instrument is not turned on.

d. Turn off printer.

e. Turn off instrument.

[rff-metcont-200]

Effective: 6-2-91

INDUCTIVELY COUPLED PLASMA - ATOMIC EMISSION SPECTROMETRIC METHOD

Scope and Application: Metals in solution can be readily analyzed by atomic emission using an inductively coupled plasma. Dissolved metals are determined in filtered and acidified samples. Total metals are determined in acidified, but unfiltered samples. Appropriate steps must be taken in all analyses to ensure that potential spectral interferences are taken into account.

Method: Inductively coupled plasma - atomic emission.

Reference: "Methods for Chemical Analysis of Water and Wastes", Method 200.7 EPA 1984.

"Inductively Coupled Plasma - Atomic Emission Spectroscopy", Method 6010, SW-846, November 1986.

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

"Instructions: Plasma 40 Emission Spectrometer", Perkin-Elmer, 1987.

Sample Handling: Acidify aqueous samples with concentrated nitric acid to pH <2. All samples must be digested prior to analysis (refer to appropriate digestion procedure). All samples must be analyzed within 6 months of sampling date.

Reagents and Appartus:

1. Plasma 40 Perkin-Elmer ICP Spectrometer
2. Argon (liquid: "high purity" or gaseous: "prepurified" grade)
3. Stock and intermediate metal standard solutions
4. EPA, ERA, or other reference standard solutions
5. Nitric acid, conc. (instra-analyzed or equivalent grade)
6. Class A volumetric glassware
7. Deionized water
8. Disposable 15 mL centrifuge tubes
9. 100 uL Eppendorf pipetter
10. 5 or 10 mL Oxford pipetter
11. Yttrium or Scandium stock solution
12. IBM AT Computer or equivalent
13. Epson 800 printer

Procedure:

Instrument Set-Up Procedure for Plasma 40:

1. Turn ON power switch if necessary (routinely left ON throughout week). Allow 1 hour for RF generator to warm up and electronic and optical components to achieve thermal equilibrium.

2. Perform daily maintenance as specified in Maintenance Procedures: check pump, pump tubing, and nebulizer tips for wear, cleanliness, etc.

3. Turn on argon at tank. The first three indicator lights on the ICP (Power, RF ready, Interlock) should light.

4. Lock pump tubing in place, raise torch to the "ignite" position, and press "RF on".

5. When plasma ignites, lower torch to the run position (the injector tip should be even with or just below the bottom of the lowest RF coil).

6. Turn on pump and aspirate rinse water*. Allow plasma to stabilize 30 to 40 minutes before starting analysis.

Computer Start-Up Procedure:

1. Turn computer and printer power on (the computer will automatically start with a memory check).

2. Type CD ICP and press Return to enter the ICP directory. Then type ICP and press Return again to load software (approximately 10-15 seconds).

3. Perform a BEC check as specified in Maintenance Procedures. The BEC and CV values must be within the specified range before any analysis is done.

Sample Analysis:

1. Before starting analysis, for each element to be analyzed:

   a. Press F1 to select the Element mode, type the appropriate element file name and press Alt F9 to retrieve it from Library.

   b. Press F8 to select Spectrum mode.

   c. Analyze a single element standard at approximately 2-10X the IDL.

   d. Analyze the ICS AB solution.

   e. Analyze 1-3 samples representative of the digestion set.

   f. Compare the displayed spectra to check for spectral interferences. Reset background correction points as needed. If there are overlapping peaks or other spectral interferences present, an alternate wavelength or interelement correction must be used.

   g. Press F8 to leave the Spectrum mode. If wavelength calibration or background correction points were changed, press F9 to save the changes.

* Rinse water should be D.I. water with a small amount of liquid detergent (such as Liquinox or Whisk) added to improve wetting of tubing and spray chamber. Approximately 1-2 mL of soap per 500 mL water should be sufficient.

2. To store a list of sample labels to be used for the analytical run, select Report mode (F3), then ID/Wt mode (F8). Enter a file name, type in N (no) in the field for raw emission counts, mg/L for uncorrected units, and leave the corrected units field blank. Enter sample labels in the sample ID field in the exact order of analysis; include all check standards, QC samples, etc. If it is possible that additional samples may be added to the end of an analytical run (dilutions, post-digestion spikes, linear range standards, etc.) add additional sample labels to the ID/Wt file in the form of single letters (A, B, C, etc.) and manually write in the correct sample labels after the analytical run is completed. Alternatively, a new ID/Wt file may be created **after** the analytical run is completed (in this case the raw data must be reprinted with the new file by selecting Report Format 1 (F5) in the report mode and responding to the prompts). Save ID/Wt files by pressing F9 (to library).

3. Press F2 to select the Method mode.

4. Type the method file name and press Alt F9 to retrieve the desired method panel from Library, or create a new panel using existing element files. Standard conditions are 35 second read delay, 2 replicates per sample, report format #2 and a data file name composed of the date (mmdd) and a sequential letter identifier (e.g. 0123B for the second analytical run on Jan 23). An internal standard (usually yttrium) must be included in any method. Background correction points are already included in each element file.

5. Add yttrium (or scandium) stock solution (1000 mg/L) as an internal standard to all standards, blanks, and samples in a ratio of 0.1 mL yttrium stock to 10 mL sample. This allows automatic correction for matrix differences in viscosity, surface tension, etc.. If the autosampler is to be used, samples can be pipetted directly into 15 mL centrifuge tubes. Otherwise mix sample and yttrium in small disposable beakers.

If autosampler is used:

1. If the autosampler is to be used, load sampler starting with the calibration standards in order of decreasing concentration (highest concentration first, calibration blank last).

2. Start automatic run (F5). Respond to the prompts that appear at the bottom of the screen:

   a. "Press start function key to begin this analysis": press F5

   b. "Enter ID/Wt file": type ID/Wt file name and press Return.

   c. "Do you wish to rinse between tubes (Y or N)": type Y and press Return. N may be selected only for clean samples where no carry-over problems are anticipated. **Always** rinse between samples when analysis is following CLP protocols, or analyzing for Sb, Cr, or Zn.

d. "Enter position of the last sample in tray": type appropriate number and press Return (you may wish to enter a number several positions past the last sample to allow room for the addition of necessary dilutions, etc. at the end of the run).

e. "Do you wish to re-standardize (Y or N)": type N or Y and press Return. N is usually selected. Y will allow restandardization of the instrument during an automatic run but additional autosampler positions will be unavailable for samples. If Y is selected, additional on-screen instructions will prompt for position of additional calibration standards.

f. "Do you wish to wavelength calibrate during the analysis? (Y or N)": type N or Y and press Return. N is usually selected. Y will allow recalibration of all wavelengths used in the current method before analysis is started but additional autosampler positions will be unavailable for samples. If Y is selected, additional on-screen instructions will prompt for position of additional wavelength calibration standards. The system will then begin the analysis.

3. When the analysis is complete press F2 to select Method mode before exiting software to ensure the data file is stored permanently. Then set up the next panel, return to Report mode to set up a new ID/Wt file or reprint data, or press ESC to exit the ICP software.

If samples are to be run manually:

1. Press F2 to start a manual run and respond to prompts to calibrate instrument: Press F6 (Standard), aspirate the first calibration standard and press Return. At the prompt, aspirate the next standard(s) and press Return. When all calibration standards have been analyzed press F5 (Blank) aspirate the calibration blank and press Return. This completes the instrument calibration.

2. To analyze samples, type in sample label if needed, press F7 (Sample), aspirate sample and press Return. Repeat with all samples in the run.

Computer Shut-Down Procedure:

1. When analysis is complete press F2 (Method mode). At the message "Do you wish to quit method"? Type Y.

2. Press "ESC". At the message "Do you wish to quit method?" Type Y.

3. Turn off computer power switch.

4. Turn off printer.

Caution: Never turn off computer power while still using ICP software. This can cause partial loss of files and other errors.

Instrument Shut-Down Procedure:

1. Aspirate a dilute nitric acid solution (approx. 10%) for 1 to 2 minutes to clean sample introduction system.

2. Aspirate D.I. water for 5 minutes to rinse system thoroughly.

3. Turn off pump and release pump tubing.

4. Press "RF off" to extinguish plasma.

5. Shut off argon flow at tank.

6. If the ICP **will not** be used for **2 days or more**, turn off ICP power switch. Otherwise, leave the ICP power **ON**.

Quality Control:

1. Establish a standard curve with the appropriate calibration standards plus a blank. Record the emission count for the internal standard in the ICP log book. The emission count should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check pump tubing, nebulizer tips, nebulizer flow, wavelength calibration, etc.).

2. **The first analyses for each analytical run are, in order:**

   a. Initial calibration verification standard (ICV)

   b. Initial calibration blank (ICB)

   c. Initial standard at 2X the CRDL (CRI). **Note:** The CRI is not necessary for Ca, Mg, Na or K.

   d. Initial interference check sample, solution A (ICSA).

   e. Initial interference check sample, solution AB (ICSAB)

   f. Laboratory control standard - an ERA, EPA, or other reference standard digested with the sample set (LCS)

To continue with sample analyses, the ICV must be within 90-110% of the true value, the ICB must be less than the CRDL, and the LCS and ICS solutions must be within 80-120% of the true value. If these QC criteria are not met, discontinue the analytical run and perform necessary troubleshooting.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are to be analyzed, a duplicate and spike are still required. Duplicates and spikes are to be within required control limits or the data must be flagged appropriately (N for spikes, * for duplicates). Additionally, if a digested spike is outside required control limits, a post-digestion spike must be analyzed for that sample.

4. For each sample batch (same matrix and project) one sample must be analyzed at an additional 5X dilution for the ICP serial dilution analysis (L). If the original sample concentration is at least 50X above the IDL, the serial dilution must agree within 10% of the original sample concentration or data for all associated samples must be flagged appropriately (E).

5. A continuing calibration verification standard (CCV) and blank (CCB) are to be analyzed, at a minimum, after every 10 analyses. If less than 10 analyses are performed, a CCV and CCB are still required. The last samples analyzed in the run are to be a CCV and CCB. The CCVs must be within 90 - 110% of the true value or the samples run after the last acceptable calibration standard are to be reanalyzed.

6. At the end of each analytical run, but before the final CCV and CCB, the CRI, and ICS solutions A and AB are to be reanalyzed. The ICS must be within 80-120% of the true value or the samples run after the last acceptable calibration verification standard are to be reanalyzed.

7. Refer to the appropriate Quality Assurance Project Plan (QAPP) for project specific QC information (additional QC requirements, matrix spike and duplicate control limits, etc.).

8. Detection limit verifications and linear range analyses must be performed each quarter. Interelement correction factors are to be determined annually at a minimum. Interelement correction factors must be recalculated on an analyte and wavelength specific basis any time background correction points are changed in an element file. Additionally, for greatest accuracy, interelement correction factors should be re-determined for any analytical batch that is expected to have high concentrations of common interferents (e.g. any soil, sediment, sludge, or leachate matrix).

Daily Maintenance Procedures - Plasma 40

1. **Pump rollers:** With the pump on, feel along the bottom of the pump to determine that all the rollers are turning smoothly with no resistance or pulling. If a "sticky" roller is found a service call must be placed to Perkin-Elmer to correct the problem. A sticky roller will cause rapid deterioration of pump tubing resulting in erratic results.

2. **Pump tubing:** Check pump tubing for excessive stretching, soft or flattened spots. This can cause irregular or diminished sample flow resulting in reduced sensitivity and lack of precision in sample results. When pump tubing is changed, (usually after 6-8 hours of use) it is necessary to trim ends of the new tubing so the length from the black stops to the end of the tubing is kept constant. Failure to trim tubing ends can cause imprecise results due to a longer sample read delay.

3. **Nebulizer tips:** Remove nebulizer end cap and check nebulizer tips visually and with the cleaning wire for clogs, salt build-up or other deposits. Follow the instructions in the Plasma 40 operating instructions for replacing nebulizer tips if necessary (Part 2, pg 3-8). Used tips may be cleaned by soaking overnight in 10% nitric acid followed by thorough rinsing with D.I. water. Finally, with the argon on, aspirate water and observe the spray pattern. The nebulizer should produce a fine, even mist with no large droplets with the direction of the spray approximately perpendicular to the face of the end cap (should not deviate more than about 20°). If the spray pattern looks uneven, "bent", or is pulsing excessively, recheck pump tubing and review nebulizer maintenance to correct the problem.

4. **BEC check:** This is an indication of how well the ICP system is performing. After the plasma has been ignited and allowed to stabilize for 30-40 minutes perform the following steps:

   a. At the DOS prompt type ICP and press Return to load software.

   b. Type MnBEC and press Alt F9 to retrieve this method from Library. Press F6 to start a manual run.

   c. Press F6 again (standards), aspirate a 1.0 mg/L Mn standard and press Return. The ICP will analyze 10 replicates of this standard. The Coefficient of Variance (CV) for these readings should be <2.0. If a higher values is obtained a sample introduction or instrument calibration problem is indicated. Check pump tubing and wavelength calibration for Mn and repeat the analysis. Record the CV in the maintenance log book.

   d. Press F5 (blank), aspirate a blank, and press Return to complete the calibration.

   f. Turn off the torch (RF off), aspirate D.I. water, and press F7 (sample). The resulting concentration should be $\leq |0.040|$. If a higher value is obtained, a problem with the sample introduction system is indicated. Review maintenance and, if the problem cannot be corrected, place a service call with Perkin-Elmer. Record the BEC in the maintenance log book.

   i. Re-light the torch and press ESC to end the manual run. Allow the plasma to stabilize 10-15 minutes before beginning any analysis.

5. **Final rinse:** When analysis for the day is complete, aspirate dilute (approx. 10%) nitric acid for one or two minutes followed by D.I. water for approximately 5 minutes. This will help prevent deposits from building up in the sample introduction system. Remember to release pump tubing when completed.

Weekly Computer Backup:

1. Once a week data files should be copied to floppy disks and deleted from the hard disk. Data files on floppy disks should be saved for one year.

2. Periodically (every 1-3 months depending on work volume), files should be reviewed, old files deleted and the entire system backed-up.

Other Maintenance:

1. Occasionally, additional maintenance will be necessary to correct problems arising from time and wear on the system. Any additional maintenance performed (including P.E. service calls) should be listed in the maintenance logs. These include periodic cleaning of the torch assembly, inspection of O-rings in torch assembly, and wavelength recalibration. Generally, these procedures will only be performed in response to observed problems. Refer to the Plasma 40 operating manual for specific directions.

ICAP CALIBRATION STANDARDS

Element	Wave-length	Detection Limit(ug/L)	Cal. Std. 1 (ug/L)	Cal. Std. 2 (ug/L)	Cal. Std. 3 (ug/L)	ICV (ug/L)	CCV (ug/L)
Al	237.335	50	20,000	400	---	2500	4000
Al	396.152	50	20,000	400	---	2500	4000
Sb	206.833	50	2000	500	250	1000	1000
Ba	233.527	10	10,000	200	---	500	2000
Be	313.107	5	1000	20	---	250	200
Cd	228.802	5	1000	50	---	500	200
Cd	214.438	5	1000	50	---	500	200
Ca	317.933	1000	200,000	10,000	---	10,000	80,000
Cr	267.716	10	10,000	500	200	1000	2000
Cr	205.552	10	10,000	500	200	1000	2000
Co	238.892	50	10,000	200	---	2500	2000
Co	228.616	10	10,000	200	---	2500	2000
Cu	324.754	10	10,000	100	---	1000	2000
Cu	224.700	20	10,000	100	---	1000	2000
Fe	238.204	20	20,000	200	---	1000	4000
Pb	220.353	100	10,000	500	---	5000	2000
Pb	216.999	100	10,000	500	---	5000	2000
Mg	285.213	1000	100,000	5000	---	10,000	40,000
Mn	257.610	10	10,000	100	---	500	2000
Ni	352.454	20	10,000	100	---	1000	2000
Ni	232.003	20	10,000	100	---	1000	2000
Ag	338.289	10	1000	---	50	500	200
Na	330.237	2000	100,000	5000	---	20,000	40,000
Sn	189.989	200	10,000	1000	---	2500	5000
V	292.402	50	10,000	500	250	2500	2000
Zn	213.856	10	10,000	100	50	500	2000

ICAP CALIBRATION STANDARDS

ICAP calibration standards are prepared from both multi-element stock solutions purchased from SPEX Industries (custom mixed standards) and single element stock solutions from VWR and Baxter (Ricca or Mallinckrodt as available).

XWE-1	XWE-2	XWE-3a	XWE-4a
2000 mg/L Fe	20,000 mg/L Ca	1000 mg/L Cr	2000 mg/L Al
1000 mg/L Cu	10,000 mg/L Mg	1000 mg/L Pb	1000 mg/L Ba
1000 mg/L Mn	10,000 mg/L Na	1000 mg/L V	1000 mg/L Co
1000 mg/L Ni		100 mg/L Cd	100 mg/L Be
1000 mg/L Zn			100 mg/L Ag

XWE-6a
500 mg/L Pb
250 mg/L Co, Al
100 mg/L Cu, Ni, Fe, Cr
50 mg/L Ba, Cd, Ag, Zn, Mn

Single Element Stock Solutions 1000 mg/L

Sb Ag
Be Na
Ca V
Cr Zn
Mg Sn

Calibration Standard #1:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, Zn:** Into a 1 L volumetric flask, add 500 mL of de-ionized (D.I.) water and 50 mL of concentrated HCl. Pipet 10 mL each of XWE-1, XWE-2, XWE-3a, and XWE-4a. Dilute to volume with D.I. water.

2. **For Sb:** Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 1.0 mL of 1000 mg/L Sb stock solution. Dilute to volume with D.I. water.

3. **For Sn:** Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 5.0 mL of 1000 mg/L Sn stock solution. Dilute to volume with D.I. water.

Calibration Standard #2:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Na, V, and Zn:** First prepare 10X dilutions each of XWE-1, XWE-2, XWE-3a, and XWE-4a. Then, into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 1.0 mL of XWE-1 (10X dilution), 5.0 mL of XWE-2 (10X dilution), 5.0 mL of XWE-3a (10X dilution), and 2.0 mL of XWE-4a (10X dilution). Dilute to volume with D.I. water.

2. **For Sb:** Into a 200 mL volumetric flask, add 100 mL of D.I. water and 10 mL of concentrated HCl. Pipet 50 mL of Sb Calibration Standard #1 and dilute to volume with D.I. water.

3. **For Sn:** Into a 200 mL volumetric flask, add 100 mL of D.I. water and 10 mL of concentrated HCl. Pipet 20 mL of Sn Calibration Standard #1 and dilute to volume with D.I. water.

Calibration Standard #3:

1. **For Cr, Ag, V and Zn:** First prepare intermediates as follows:

   50 mg/L Ag and Zn: Into a 100 mL volumetric flask, add 10 mL of 1:1 HCl. Pipet 5.0 mL each of single element Ag and Zn stock solutions and dilute to volume with D.I. water.

   100 mg/L Cr: Into a 100 mL volumetric flask pipet 10.0 mL of single element Cr stock solution. Add 5 mL of 1:1 HCl and dilute to volume with D.I. water.

   50.0 mg/L V: Into a 100 mL volumetric flask pipet 5.0 mL of single element V stock solution. Add 5 mL of 1:1 HCl and dilute to volume with D.I. water.

   Then, into a 500 mL volumetric flask, add 250 mL D.I. water and 25 mL of concentrated HCl. Pipet 1.0 mL of 100 mg/L Cr intermediate, 0.5 mL of 10 mg/L Ag-Zn mixed intermediate, and 2.5 of 50 mg/L V intermediate. Dilute to volume with D.I. water.

2. **For Sb:** Into a 100 mL volumetric flask, add 50 mL of D.I. water and 10 mL of 1:1 HCl. Pipet 50 mL of Calibration Standard #2 and dilute to volume with D.I. water.

Initial Calibration Verification:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, and Zn:** Into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 10 mL of XWE-6a, 10 mL of 1000 mg/L Mg stock, 10 mL of 1000 mg/L Ca stock, and 20 mL of 1000 mg/L Na stock solutions. Dilute to volume with D.I. water.

2. **For Sb:** Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 0.5 mL of 1000 mg/L Sb stock and dilute to volume with D.I. water.

3. **For Sn:** Into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 2.5 mL of 1000 mg/L Sn stock and dilute to volume with D.I. water.

Continuing Calibration Verification Standard:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, and Zn:** Into a 1 L volumetric flask, add 500 mL D.I. water and 50 mL of concentrated HCl. Pipet 2.0 mL each of XWE-1, XWE-3a, XWE-4a, and 4.0 mL of XWE-2. Dilute to volume with D.I. water.

2. **For Sb:** Use Sb Initial Calibration Verification Standard.

3. **For Sn:** Into a 200 mL volumetric flask, add 100 mL D.I. water and 10 mL of concentrated HCl. Pipet 1.0 mL of 1000 mg/L Sn stock and dilute to volume with D.I. water.

Effective: 7-2-91

ACID DIGESTION FOR AQUEOUS SAMPLES AND EXTRACTS
ICP/Flame-AA

Scope and Application: This acid digestion is applicable to all aqueous sample matrices. A nitric/hydrochloric acid digestion is used to prepare all samples which are to be analyzed by flame atomic absorption spectroscopy (flame-AA) or by inductively coupled plasma spectroscopy (ICP). A nitric acid/hydrogen peroxide digestion is used to prepare samples for analysis by graphite furnace atomic absorption spectroscopy (GFAA).

Method: Nitric acid/hydrogen peroxide and nitric/hydrochloric acid digestions

Reference: "Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Sample Handling: Aqueous samples must be acidified with concentrated nitric acid to pH <2. Set up digestion as soon as possible; digested sample must be analyzed within 6 months.

Reagents and Apparatus:

1. Hot plate
2. 250 mL beakers
3. 100 mL graduated cylinders
4. Class A volumetric glassware
5. Deionized (D.I.) water
6. Instra-analyzed nitric acid, or equivalent
7. Distilled nitric acid (GFAA digestion only)
8. Instra-analyzed HCl acid, or equivalent
9. Stock and standard metal solutions
10. Whatman #42 filter paper
11. Glass or plastic funnels
12. Watch glasses
13. 30% Hydrogen peroxide

Reagent Preparation:

1. **Intermediate and working metal solutions:** Refer to the specific metal SOP for instructions on preparation.

2. **1:1 Hydrochloric acid (HCl):** Using a graduated cylinder, add 250 mL D.I. water to a to a 500 mL (or 1 L) repipettor. Carefully add 250 mL of concentrated HCl and mix.

3. **1:1 Nitric acid (HNO_3):** Using a graduated cylinder, add 250 mL D.I. water to a to a 500 mL (or 1 L) repipettor. Carefully add 250 mL of concentrated HNO_3 and mix.

Notes:

1. A separate digestion is required for mercury analyzed by the AA-Cold Vapor technique. (See "Mercury Digestion-Aqueous Samples")

2. All samples, duplicates, and spikes, as well as any required prep or digested blanks and standards, must be carried through the digestion procedure.

3. If samples boil or go to dryness (**any** dry spots on the bottom of the beaker) at any time during the digestion, some of the analyte may have been lost. The digestion must be discarded and the affected samples must be reprepared.

4. If elevated analyte levels are expected, the spike concentration may be increased accordingly.

Procedure:

Digestion Procedure for Flame-AA and ICP:

1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with D.I. water prior to use.

2. Measure out 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.

3. Add 2.0 mL of 1:1 HNO_3 and 10 mL of 1:1 HCl.

4. Cover with a watch glass and heat on the hot plate for 2 hours or until the volume has been reduced to between 25 and 50 mL. Adjust the temperature of the hot plate as needed to prevent samples from boiling.

5. Allow samples to cool. If any insoluble material remains, filter samples through Whatman #42 filters. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Rinse beakers and filters with D.I. water and dilute to volume to 100 mL.

6. Samples are now ready for analysis using the AA-flame or ICP methods.

Digestion Procedure for GFAA:

1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with D.I. water prior to use.

2. Measure out 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.

3. Add 1.0 mL of 1:1 HNO_3 and 2.0 mL of 30% H_2O_2.

4. Cover with a watch glass and heat on the hot plate for 2 hours or until the volume has been reduced to between 25 and 50 mL. Adjust the temperature of the hot plate as needed to prevent samples from boiling.

5. Allow samples to cool. If any insoluble material remains, filter samples through Whatman #42 filters. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Rinse beakers and filters with D.I. water and dilute to volume to 100 mL.

6. Samples are now ready for GFAA analysis.

Quality Control:

1. A digested blank and standard (spiked blank) must be included with each batch of samples that is digested. The blank is a check for possible contamination during the digestion process; the standard is a check for possible analyte loss during digestion.

2. A matrix spike and duplicate must be prepared, at a minimum, for every 10 samples digested. If fewer than 10 samples are digested a spike and duplicate are still required.

Effective Date: 7-2-91

ANTIMONY - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 204.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.005 mg/L

Optimum Range: 0.005 - 0.100 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	14
Slit Width (nm):	0.2
Slit Height:	Normal
Wavelength (nm):	217.6
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	2.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	1.40

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	85	5.0	3.0	NORMAL	NO
2	95	25.0	3.0	NORMAL	NO
3	120	10.0	3.0	NORMAL	NO
4	120	5.0	3.0	NORMAL	NO
5	900	10.0	3.0	NORMAL	NO
6	900	5.0	3.0	NORMAL	NO
7	900	2.0	0.0	NORMAL	NO
8	2300	1.0	0.0	NORMAL	YES
9	2300	2.0	0.0	NORMAL	YES
10	2300	2.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix Modifier Volume: 5 uL (0.25% Nickel Nitrate).

Calibration standards: 25.0, 50.0, 100.0 ug/L.

Graphite Tube Type: Pyrolytic coated partition tube.

Sample Handling: Acidify with nitric acid to pH < 2. Analyze within 6 months.

Reagent Preparation:

1. **Standard Antimony Solution (1000 ug/L Antimony):** Pipet 1.00 mL of the 1000 ppm stock antimony solution into a 1000 mL volumetric flask, add 0.5 mL HNO_3 and dilute to volume with D.I. water. Prepare fresh daily.

2. **Calibration standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Antimony Standard	Dilute to
25.0 ug/L	2.5 mL of 1000 ug/L Sb	100 mL
50.0 ug/L	5.0 mL of 1000 ug/L Sb	100 mL
100 ug/L	10 mL of 1000 ug/L Sb	100 mL

3. **Nickel Nitrate 0.25% Solution:** In a 100 mL volumetric flask dissolve 1.25g of $Ni(NO_3)_2 \cdot 6H_2O$ in D.I. water and dilute to 100 mL. Prepare fresh every 6 months.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of background correction is required.

4. The use of halide acids should be avoided.

5. Nickel nitrate is added as a matrix modifier to control interferences.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

If antimony is to be analyzed in the concentration mode, use the 25.0, 50.0, and 100 ug/l standards for instrument calibration and follow the procedure for analysis in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable range(90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required. (See the Furnace Decision Tree for more detail.)

6. An EPA reference standard will be analyzed with each analysis.

Calculations:

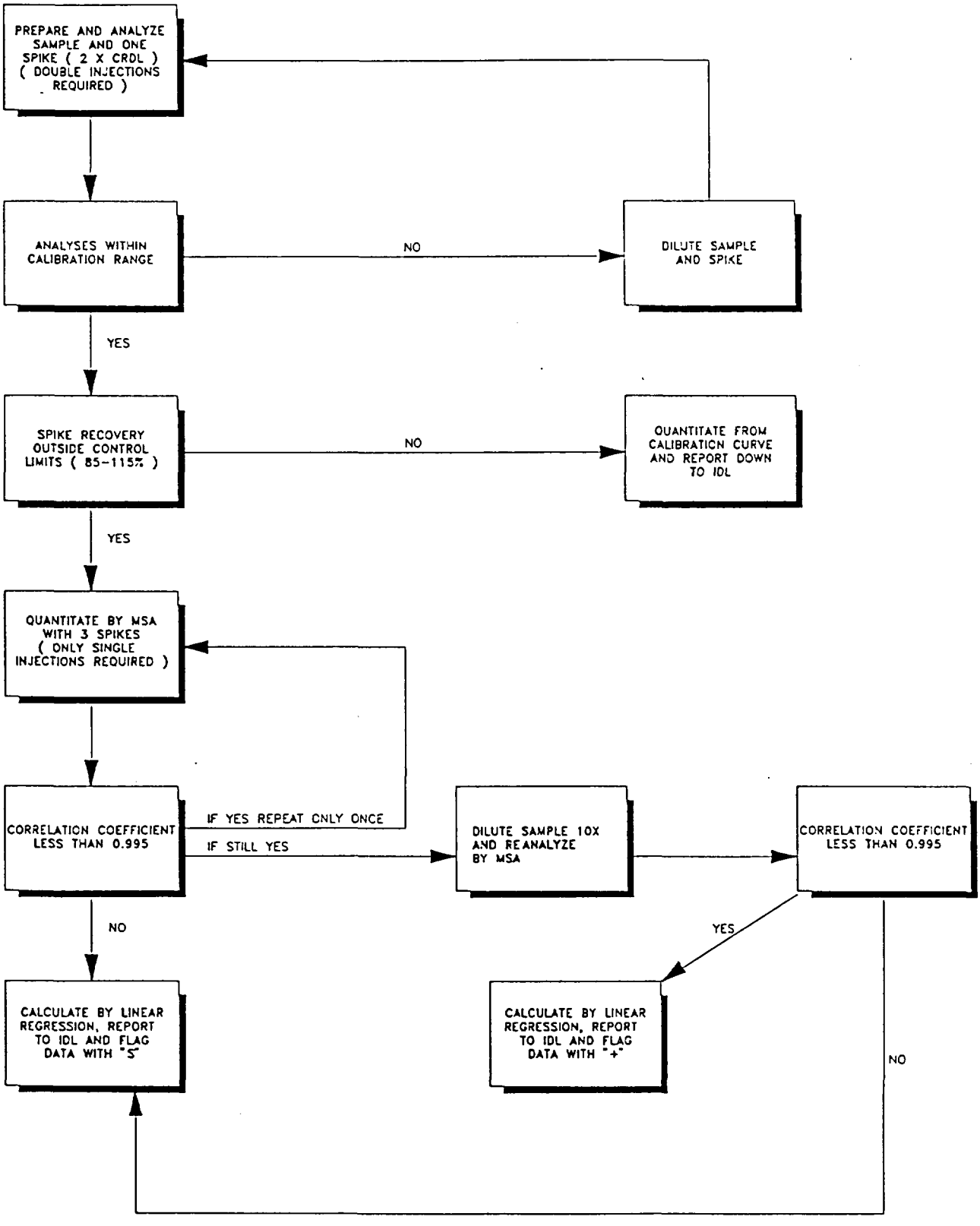
1. Calculate using the instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-298]

WARZYN

FURNACE CLP DECISION TREE



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Effective Date: 7-2-91

ARSENIC - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 206.2

"Analytical Methods for Zeeman Graphite Tube Atomizers"-Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Sample Handling: Acidify with nitric acid to pH <2. Analyzed within 6 months. All samples must be digested prior to analysis.

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	10
Slit Width (nm):	1.0
Slit Height:	Normal
Wavelength (nm):	193.7
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	0.95

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	220	5.0	3.0	NORMAL	NO
3	240	40.0	3.0	NORMAL	NO
4	240	5.0	3.0	NORMAL	NO
5	1400	5.0	3.0	NORMAL	NO
6	1400	10.0	3.0	NORMAL	NO
7	1400	1.0	0.0	NORMAL	NO
8	2600	0.8	0.0	NORMAL	YES
9	2600	2.0	0.0	NORMAL	YES
10	2600	1.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix modifier volume: 5 uL (0.25% nickel nitrate).

Calibration standards: 10.00, 20.00, 50.00 ug/L.

Graphite Tube Type: Pyrolytic coated plateau tube

Reagent Preparation:

1. **Standard Arsenic Solution (1000 ug/L Arsenic):** Pipet 1.00 mL of the 1000 ppm stock arsenic solution into a 1000 mL volumetric flask, add 0.5 mL HNO_3 and dilute to the mark with deionized water. Prepare fresh monthly.

2. **Calibration standards:** Digest according to the appropriate digestion procedure. Prepare fresh monthly.

Concentration of Standard	Volume of Arsenic Standard	Dilute to
0 ug/L	0 mL of 1000 ug/L As	100 mL
10 ug/L	1 mL of 1000 ug/L As	100 mL
20 ug/L	2 mL of 1000 ug/L As	100 mL
50 ug/L	5 mL of 1000 ug/L As	100 mL

3. Nickel Nitrate (0.25%): In a 100 mL volumetric flask dissolve 1.25 g of $Ni(NO_3)_2 \cdot 6H_2O$ in D.I. water and dilute to 100 mL. Prepare fresh every 6 months.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. Nickel nitrate is added as a matrix modifier to minimize volatilization losses during the drying and charring steps.

4. The use of background correction is required.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

If Arsenic is to be analyzed in concentration mode, use the 10.0, 20.0, and 50.0 ug/L standards for instrument calibration, and follow the procedures for analysis in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable range (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit of 10 ug/L after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required. (See the Furnace Decision Tree for more detail.)

6. An EPA reference sample will be analyzed with each analysis.

Calculations:

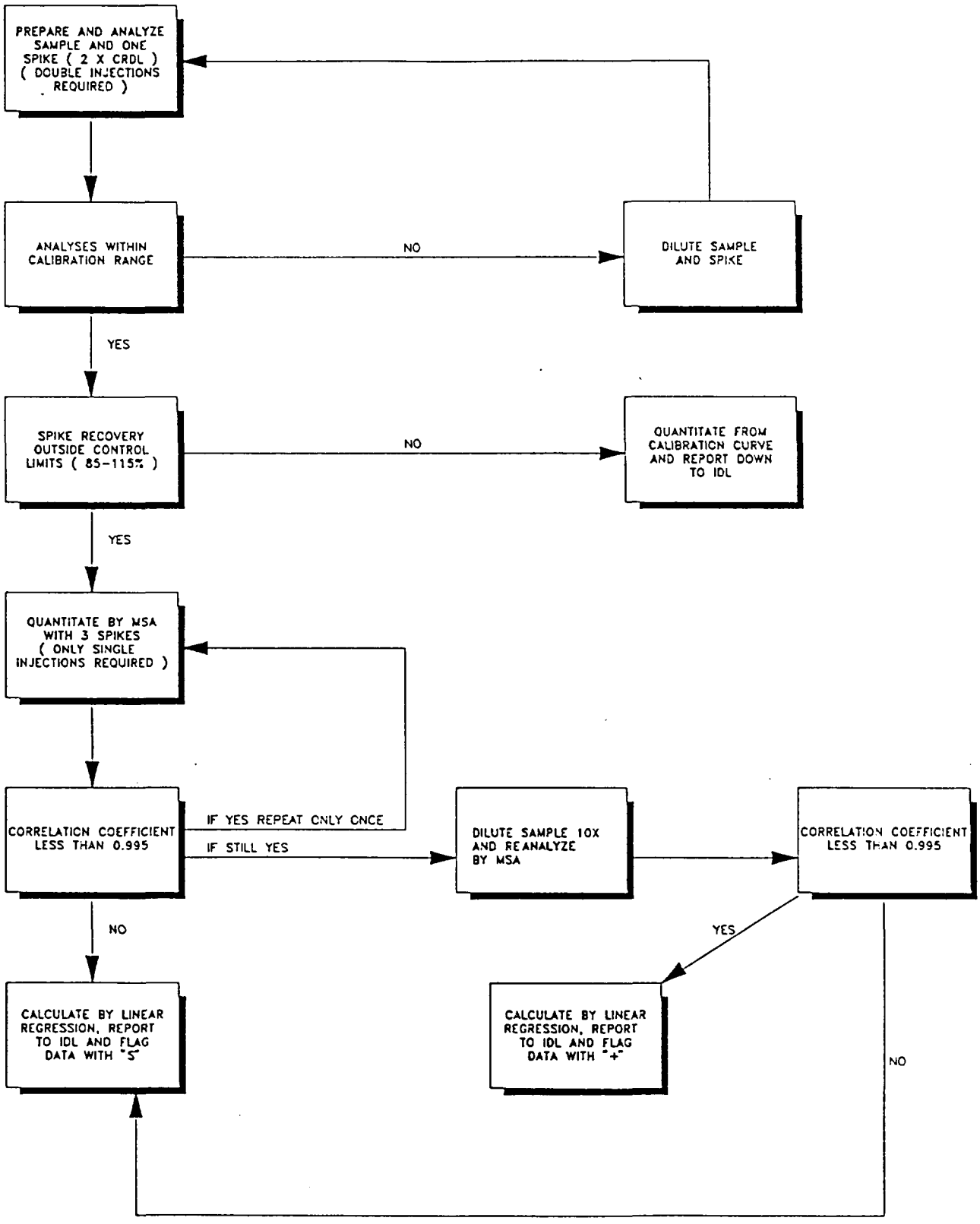
1. Calculate using the instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-297]

WARZYN

FURNACE CLP DECISION TREE



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Effective Date: 7-2-91

CADMIUM - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 213.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.0002 mg/L

Optimum Range: 0.0002 - 0.0030 mg/L

Sample Handling: Acidify with nitric acid to pH <2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	3
Slit Width (nm):	0.5
Slit Height:	Normal
Wavelength (nm):	228.8
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	0.70

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	230	5.0	3.0	NORMAL	NO
3	260	40.0	3.0	NORMAL	NO
4	260	5.0	3.0	NORMAL	NO
5	700	5.0	3.0	NORMAL	NO
6	700	5.0	3.0	NORMAL	NO
7	700	1.0	0.0	NORMAL	NO
8	2000	0.8	0.0	NORMAL	YES
9	2000	2.0	0.0	NORMAL	YES
10	2000	2.0	3.0	NORMAL	NO

Sample Volume: 12 uL

Matrix Modifier Volume: 4 uL (Monobasic ammonium phosphate)

Calibration standards: 1.00, 2.00, 3.00 ug/L

Graphite Tube Type: Pyrolytic coated plateau tube

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard Cadmium Solution (1000 ug/L Cadmium):** Pipet 1.00 mL of the 1000 ppm stock cadmium solution into a 1000 mL volumetric flask, add 1/2 mL HNO_3, and dilute to the mark with D.I. water. Prepare fresh daily.

2. **Working Cadmium Solution (100 ug/L Cadmium):** Pipet 10 mL of the 1000 ug/L cadmium into a 100 mL volumetric flask and dilute to the mark with D.I. water. Prepare fresh daily.

3. Standards (Prepare fresh daily.):

Concentration of Standard	Volume of Cadmium Standard	Dilute to
1.00 ug/L	1 mL of 100 ug/L Cd	100 mL
2.00 ug/L	2 mL of 100 ug/L Cd	100 mL
3.00 ug/L	3 mL of 100 ug/L Cd	100 mL

4. **Monobasic Ammonium Phosphate Solution (5000 mg/L):** Add 1.0 g of ammonium phosphate (monobasic) to a 100 mL volumetric flask. Dissolve in D.I. water and dilute to volume.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of background correction is required.

4. The cadmium flame or ICP procedure is recommended where concentrations are greater than 0.10 mg/L.

5. Ammonium phosphate is added as a matrix modifier to improve peak shape and allow higher ashing temperatures.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

Use of peak area is required.

If cadmium is to be analyzed in concentration mode, use the 1.00, 2.00, and 3.00 ug/L standards for instrument calibration and follow the procedures for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable range (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference sample will be analyzed with each analysis.

Calculations:

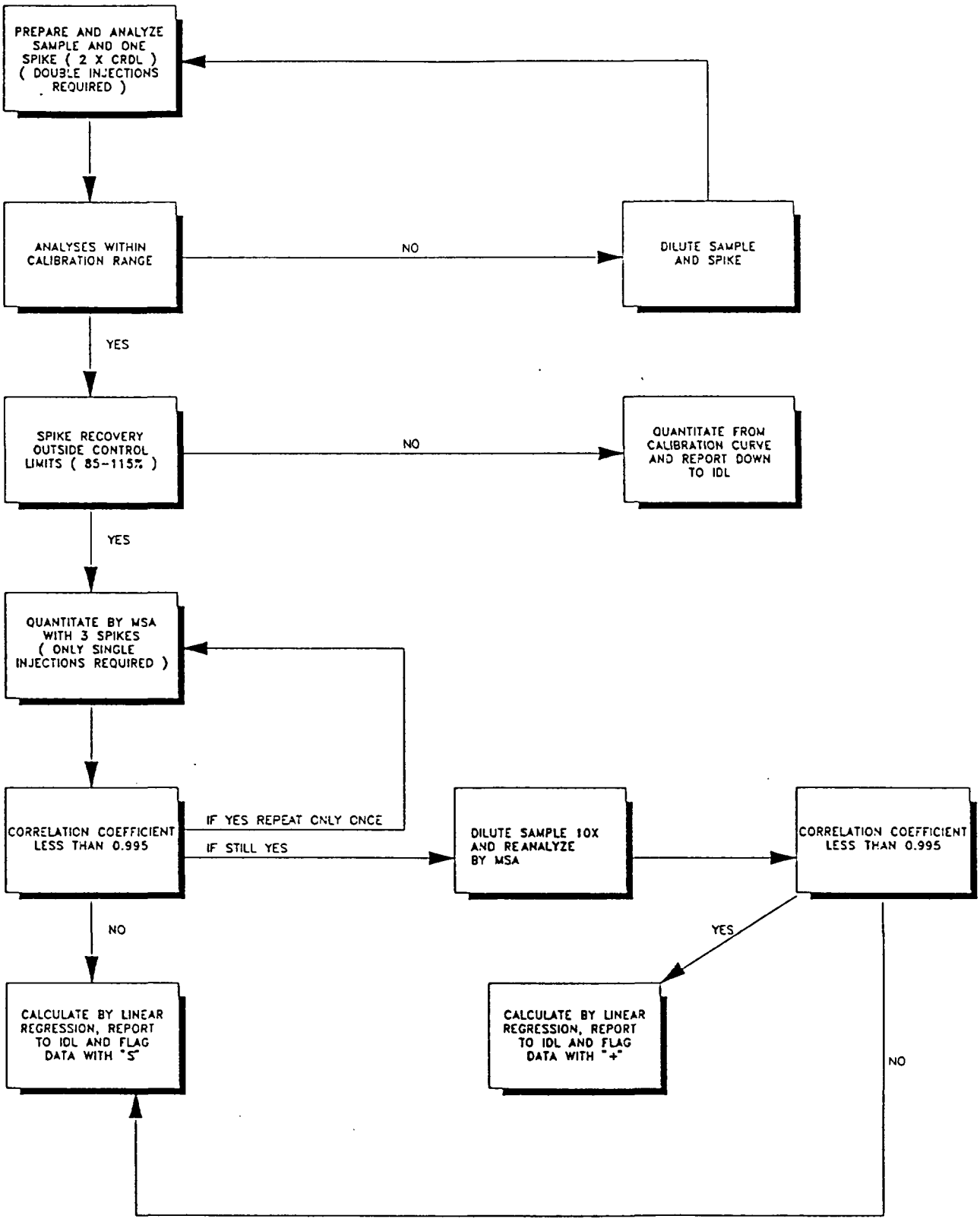
1. Calculate using instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-292]

WARZYN

FURNACE CLP DECISION TREE



Effective Date: 1-14-92

LEAD - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 239.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986.

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Sample Handling: Acidify with nitric acid to pH < 2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	4
Slit Width:	0.5
Slit Height:	Normal
Wavelength	283.3
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background:	On
Maximum Absorbance:	1.40

Furnace Parameters:

Step	Temp (*C)	Time (sec)	Gas Flow (L/Min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	220	5.0	3.0	NORMAL	NO
3	240	45.0	3.0	NORMAL	NO
4	240	5.0	3.0	NORMAL	NO
5	650	5.0	3.0	NORMAL	NO
6	650	15.0	3.0	NORMAL	NO
7	650	1.0	0.0	NORMAL	NO
8	2200	0.9	0.0	NORMAL	YES
9	2200	2.0	0.0	NORMAL	YES
10	2500	2.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix modifier volume: 5 uL 0.5% w/v Ammonium Phosphate Monobasic or 5 ul of lanthanum nitrate modifier.

Calibration standards: 3.0, 10.0, 20.0, 50.0 ug/L

Graphite Tube Type: Pyrolytic Coated Plateau Tube

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard lead solution (10.0 mg/L Lead):** Pipet 1.0 mL of the 1000 ppm stock lead solution into a 100 mL volumetric flask, add 0.5 mL HNO_3 and dilute to volume with deionized water. Prepare fresh daily.

2. **Standard lead solution (100ug/L Lead):** Pipet 1.0 mL of the 10.0 mg/L lead standard into a 100 mL volumetric flash, add 0.5 mL HNO_3 and dilute to volume with deionized water. Prepare fresh daily.

3. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Lead Standard	Dilute to
3.0 ug/L	3 mL of 100 ug/L Pb	100 mL
10.0 ug/L	10 mL of 100 ug/L Pb	100 mL
20.0 ug/L	20 mL of 100 ug/L Pb	100 mL
50.0 ug/L	50 mL of 100 ug/L Pb	100 mL

4. **Ammonium phosphate matrix modifier:** Dissolve 0.5g ammonium phosphate monobasic in 100mL D.I. water.

5. **Lanthanum nitrate matrix modifier:** Dissolve 5.864g of La_2O_3 in 10 ml concentrated nitric acid and dilute to 1 L with D.I. water.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of background correction is required.

4. Ammonium phosphate is added as a matrix modifier to improve peak shape and allow higher ashing temperatures. Ammonium phosphate is the preferred matrix modifier for groundwater, residential wells, and any other samples where chloride or sulfate concentrations are expected to be less than 100 mg/L. Due to its more corrosive nature, lanthanum nitrate should be used as matrix modifier only if chloride and/or sulfate concentrations are expected to exceed 100 mg/L.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

If lead is to be analyzed in the concentration mode, use the 3.0, 10.0, 20.0 and 50.0 ug/L standards for instrument calibration and follow the procedure for analyzing using the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard of 20.0 ug/L and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. **Criteria for standard additions are:**

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required. (**See Furnace Decision Tree for more detail.**)

6. An EPA reference sample will be analyzed with each analysis.

Calculations:

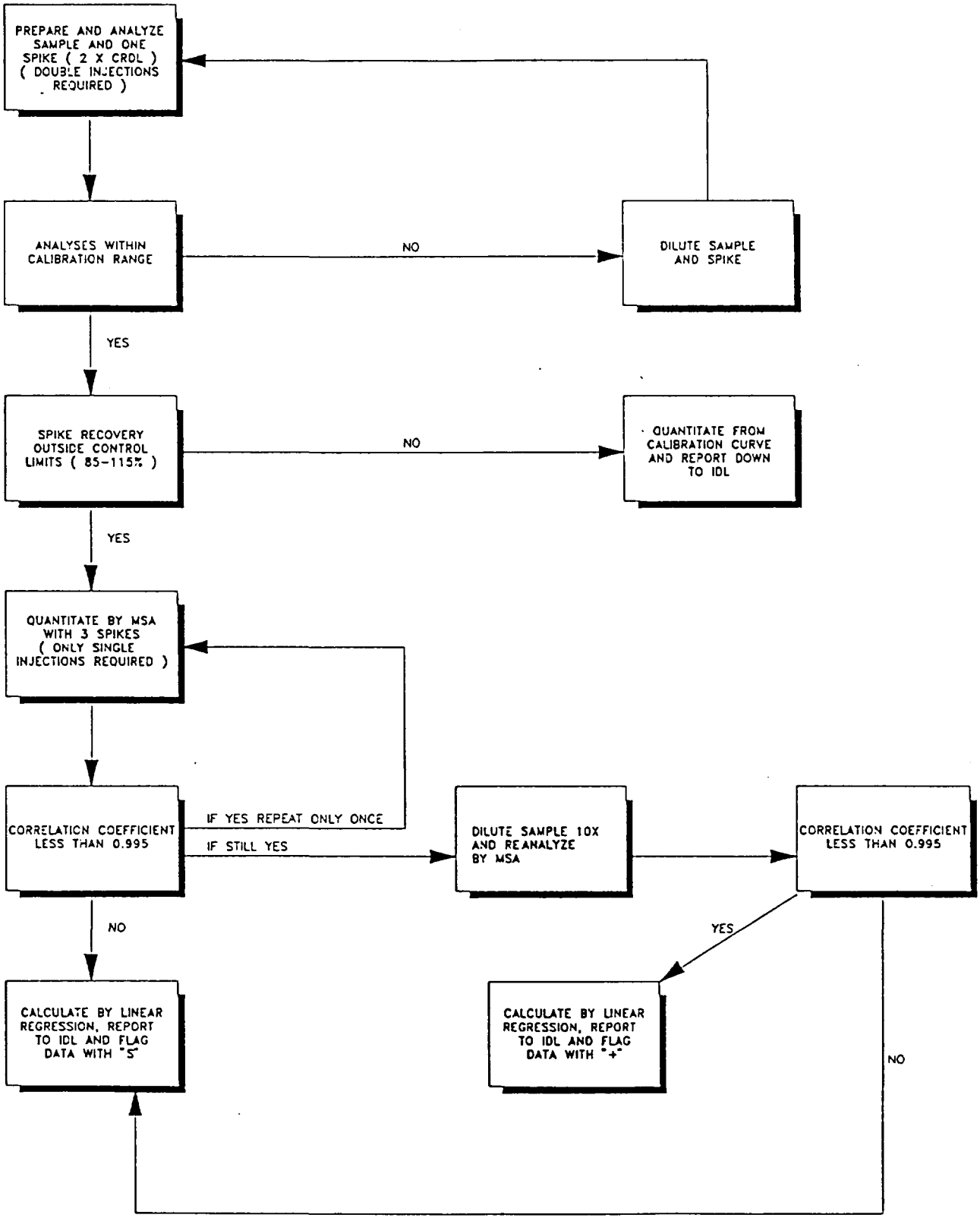
1. Calculate using the instrument concentration mode, or

3. For method of standard additions calculate using linear regression.

[rff-metcont-284]

WARZYN

FURNACE CLP DECISION TREE



Effective: 7-2-91

MERCURY DIGESTION LIQUID SAMPLES

Scope and Application: This mercury digestion method is applicable to drinking, surface, groundwater, domestic, and industrial wastewaters.

Method: Nitric/sulfuric acid digestion

Reference: EPA 1983, Method 245.1
"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Sample Handling: Preserve with concentrated HNO_3 to pH <2. Analyze within 28 days of sampling.

Reagents and Apparatus:

1. Water bath set @ 95°C
2. BOD bottles; 300 mL
3. Class A volumetric glassware
4. Instra-analyzed sulfuric acid
5. Instra-analyzed nitric acid
6. Potassium persulfate
7. Potassium permanganate
8. Sodium chloride
9. Hydroxylamine hydrochloride solution
10. Various Class A volumetric pipettes
11. Mercury stock and standard solutions

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. **Sodium chloride-hydroxylamine hydrochloride solution:** In a 1000ml volumetric flask dissolve 120.0 g of sodium chloride and 120.0 g of hydroxylamine hydrochloride in D.I. water, dilute to 1 liter.

2. **Potassium permanganate (5% solution, w/v):** In a 1000ml volumetric flask dissolve 50.0 g of potassium permanganate in D.I. water, dilute to 1 liter.

3. **Potassium persulfate (5% solution, w/v):** In a 1000ml volumetric flask dissolve 50.0 g of potassium persulfate in D.I. water, dilute to 1 liter.

4. **Intermediate mercury standard (10.0 mg/L):** Transfer 1.0 mL stock mercury (1000 mg/L) solution, plus 0.5 mL nitric acid, into a 100 mL volumetric flask and dilute to the mark with D.I. water. Prepare fresh daily!

5. **Working mercury standard (0.100 mg/L):** Transfer 1.0 mL of the 10.0 mg/L intermediate standard, plus 0.5 mL nitric acid, into a 100 mL volumetric flask and dilute to the mark with D.I. water. Prepare fresh daily!

Notes:

1. The mercury standards are volatile and unstable. Standards must be prepared daily.

2. Because of the toxic nature of mercury vapor, precaution must be taken to avoid inhalation. Vent the mercury vapor into an exhaust hood or pass the vapor through an absorbing media.

3. Hydroxylamine sulfate may be used rather than hydroxylamine hydrochloride.

4. All blanks, standards, and samples must be carried through the digestion procedure.

5. **Interferences:**

   a. Potassium permanganate is added to eliminate interferences from sulfide. Concentrations as high as 20 mg/L sulfide as sodium sulfide do not interfere.

   b. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on recovery of mercury from spiked samples.

   c. Seawaters, brines, and industrial effluents, high in chlorides, will require additional potassium permanganate. Care must be taken to ensure that the same amount of potassium permanganate is added to all samples, blanks, and standards so total volume remains constant.

Procedure:

All glassware is to be washed with soap and water, rinsed with tap water, acid rinsed with 10% HNO_3, and final rinsed with D.I. water.

Standard Preparation:

1. **The standard curve is to consist of the following standards:**

Standard Concentration
0.00 ug/L
0.50 ug/L
1.00 ug/L
5.00 ug/L
10.0 ug/L

2. Pipet 0, 0.5, 1.0, 5.0, and 10.0 mL aliquots of 0.10 mg/L working stock mercury solution to 300 mL BOD bottles.

3. Add D.I. water to bring volume to 100 mL and continue with the digestion procedure.

Sample Preparation:

1. Transfer 100 mL, or an aliquot diluted to 100 mL, to a 300 mL BOD bottle.

   To Spike: Pipette 1.0 mL of 0.10 mg/L mercury standard into the sample bottle.

Digestion:

1. Add 5 mL conc. sulfuric acid and 2.5 mL conc. nitric acid to each bottle. Mix by swirling.

2. Add 15 mL potassium permanganate solution to each bottle, mix by swirling. Allow to stand for at least 15 minutes. If the bottle does not remain purple in color, additional potassium permanganate is required. Equal volumes of potassium permanganate must be added to all bottles.

3. Add 8 mL of potassium persulfate solution to each bottle and heat for 2 hours in a water bath maintained at 95°C. Check the bottles periodically throughout the 2 hours to insure the samples remain purple. Add additional potassium permanganate, if needed, to all bottles in the digestion set.

4. Cool to room temperature.

5. Samples are now ready for analysis using the AA-cold vapor procedure.

Quality Control:

1. Refer to the cold vapor SOP for quality control requirements.

[rff-metcont-222]

Effective: 7-2-91

TOTAL MERCURY - AUTOMATED

Scope and Application: This method is applicable to digested drinking, surface, groundwater, domestic, and industrial wastewaters, soils, and sediments. All samples must be digested prior to analysis.

Method: Automated Cold Vapor

Reference: EPA 1984, Method 245.1, 245.5
SW846, 1982, Method 7471
"Vapor generation Accessory Operation Manual", Varian, 1984
"Statement of Work for Inorganic Analysis, No. 788", EPA 1989

Detection Limits: 0.20 ug/L

Optimum Range: 0.20-10.0 ug/L

Sample Handling: Samples should be kept capped until just prior to analysis.

Instrument Conditions:

Instrument Mode:		Absorbance
Calibration Mode:		Concentration
Measurement Mode:		Integration
Lamp Position:		1
Lamp Current (mA):		4
Slit Width (nm):	0.5	
Wavelength (nm):	253.7	
Flame:		Air only
Sample Introduction:		Auto Normal
Delay Time:		60
Time Constant:		0.05
Measurement Time (sec):		3.0
Replicates:		3
Background Correction:		On
Air Flow:	0.00	
Rinse Rate:		1
Rinse Time:		5.0
Recalibration Rate:		0
Reslope Rate:	0	

Reagents and Apparatus:

1. Varian SpectrAA20
2. Varian VGA-76 (cold vapor generator)
3. Varian PSC-56 (autosampler)
4. Sodium chloride
5. Hydroxylamine hydrochloride
6. Stannous chloride
7. Hydrochloric acid
8. Mercury lamp
9. Tygon tubing
10. Whatman #4 filter paper or equivalent

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. **Hydrochloric acid (20% v/v):** Add 100 mL of conc. HCl to 200 mL D.I. water in a 500 mL volumetric flask, dilute to 500 mL. **Prepare in the hood!**

2. **Stannous chloride (25% w/v):** Dissolve 125.0 g stannous chloride in 500 mL of 20% HCL. Prepare fresh every month.

3. **Sodium chloride-hydroxylamine hydrochloride solution:** Dissolve 120.0 g of sodium chloride and 120.0 g of hydroxylamine hydrochloride in D.I. water, dilute to 1 liter.

Notes:

1. Because of the toxic nature of mercury vapor, precaution must be taken to avoid inhalation. Vent the mercury vapor into an exhaust hood or pass the vapor through an absorbing media.

2. A 10% solution of stannous sulfate may be substituted for stannous chloride.

3. Hydroxylamine sulfate may be used rather than hydroxylamine hydrochloride.

4. **Interferences:**

   a. Potassium permanganate is added to eliminate interferences from sulfide. Concentrations as high as 20 mg/L sulfide as sodium sulfide do not interfere.

   b. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on recovery of mercury from spiked samples.

   c. Seawaters, brines, and industrial effluents, high in chlorides, will require additional potassium permanganate. during the oxidation step, chlorides are converted to free chlorine which also absorbs at the same wavelength as mercury.

d. Certain volatile organic materials that absorb at this wavelength may also cause an interference. A preliminary run without reagents can determine if this type of interference is present.

5. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by leaving digested mercury samples uncapped in the hood for approximately 30 minutes after the addition of the sodium chloride hydroxylamine solution, or allowing a prepared autosampler tray to stand 10-20 minutes before starting an automated analytical run.

6. If particulates remaining in the digested sample cause obstructions in the autosampler tubing, samples can be filtered through Whatman #4 filter paper, or its equivalent, after excess permanganate has been reduced.

Procedure:

For instrument set-up procedures, refer to the Atomic Absorption Spectrometry, Flame section of this manual.

For concentration mode, use 0.5, 1.0, 5.0 and 10.0 ug/L standards to calibrate the instrument.

A. **Cold Vapor System Set-up:**

1. Insert quartz cell in burner chamber. (Attaches to the air/acetylene burner head.)

2. Visually align cell, checking light path with a white card or paper.

3. Select "Optimization" page and adjust cell for maximum signal.

4. Replace pump tubing on vapor generator.

5. Fill reagent bottles with D.I. water and 25% SnCl (stannous chloride) solution as labelled.

B. **Sample Analysis:**

1. Prior to analysis, add 6 mL of the sodium chloride-hydroxylamine solution to each bottle to reduce the excess permanganate. Additional sodium chloride-hydroxylamine may be needed to discharge the purple color; equal volumes must be added to all bottles in the digestion set.

2. Pour approximately 12 mL of samples, standards, and blanks into sample tubes and arrange on the autosampler.

3. Turn on argon supply (46 psi recommended).

4. Turn on autosampler power.

5. Turn on vapor generator power (peristaltic pump will run continuously while power is on. Check reagent levels periodically during long runs).

6. Allow pump to operate for 3 to 4 minutes to stabilize flow rates.

7. Start automatic run.

8. Run will stop automatically after it is completed. Press "stop" to release computer.

9. Pull tubing ends out of reagents and let pump empty the lines. Power off pump and release pump tubing. Turn off argon supply.

10. Power off autosampler, printer and AA if done with analyses for the day.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, tubing, lamp alignment, pump, etc.)

2. A quality control calibration check standard of 5.0 ug/L and a blank are to be analyzed initially and, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a check standard and a blank are still required. The last samples analyzed in the run are to be the check standard and blank. These standards must be within acceptable ranges (80-120%) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges or the data will be flagged appropriately.

4. An EPA or ERA reference standard will be analyzed with each analytical run. The reference standard must be within acceptable limits (80-120% of the true value) before any samples are analyzed.

Calculation:

1. Calculate using the instrument concentration mode, or

2. For method of standard additions, use linear regression.

[rff-metcont-225]

Effective: 7-2-91

POTASSIUM - VARIAN 20

Method: Flame Emission: Direct Aspiration

Reference: "Analytical Methods for Flame Spectrophotometry, Varian 1979.

"Standard Methods for the Examination of Water and Wastewater", 16th Edition, Method 322B, 1985.

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.10 mg/L

Optimum Range: 0.10 - 10.0 mg/L

Sample Handling: Acidify with nitric acid to pH <2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

1. Instrument mode: Emission
2. Wavelength: 766.5 nm
3. Slit Width: 1.0
4. Fuel: Acetylene
5. Oxidant: Air
6. Type of flame: Oxidizing, lean, blue
7. Standards to use for calibration: 0.50, 1.00, 2.00, 5.00, 10.0 mg/L.

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard Potassium Solution (100 mg/L Potassium):** Pipet 10 mL of the 1000 ppm stock potassium solution into a 100 mL volumetric flask, add 0.5 mL HNO_3, and dilute to volume with D.I. water.

2. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Potassium Standard	Dilute to
0.50 mg/L	0.5 mL of 100 mg/L	100 mL
1.00 mg/L	1 mL of 100 mg/L	100 mL
2.00 mg/L	2 mL of 100 mg/L	100 mL
5.00 mg/L	5 mL of 100 mg/L	100 mL
10.0 mg/L	10 mL of 100 mg/L	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Flame - Direct Aspiration section of this manual.

If potassium is to be analyzed in concentration mode, use the 1.00, 5.00, and 10.0 mg/L standards to calibrate the instrument and follow the procedure for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The emission readings should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, flame head alignment, etc.).

2. A quality control calibration standard of 1.00 mg/L and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

4. An EPA reference sample will be analyzed with each analysis.

Calculations:

1. Calculate using the instrument concentration mode, or

2. For method of standard additions, use linear regression.

[rff-metcont-280]

Effective: 7-2-91

SELENIUM - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 270.2

"Analytical Methods for Zeeman Graphite Tube Atomizer"-Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Height
Lamp Current (mA):	18
Slit Width (nm):	1.0
Slit Height:	Normal
Wavelength (nm):	196.0
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	1.20

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	220	5.0	3.0	NORMAL	NO
3	240	40.0	3.0	NORMAL	NO
4	240	5.0	3.0	NORMAL	NO
5	1400	5.0	3.0	NORMAL	NO
6	1400	10.0	3.0	NORMAL	NO
7	1400	1.0	0.0	NORMAL	NO
8	2600	0.8	0.0	NORMAL	YES
9	2600	2.0	0.0	NORMAL	YES
10	2600	1.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix Modifier Volume: 5 uL (0.25% nickel nitrate)

Standards to use for curve set-up: 5.0, 10.0, 20.0, 50.0 ug/L.

Graphite Tube Type: Pyrolytic coated plateau tube

Sample Handling: Acidify with nitric acid to pH < 2. Analyze within 6 months.

Reagent Preparation:

1. **Standard selenium solution (1000 ug/L Selenium):** Pipet 1.00 mL of the 1000 ppm stock selenium solution into a 1000 mL volumetric flask, add 0.5 mL HNO_3 and dilute to volume with D.I. Prepare fresh daily.

2. **Calibration standards:** Digest standards according to the appropriate digestion procedure. Prepare fresh monthly.

Concentration of Standard	Volume of Selenium Standard	Dilute to
5.0 ug/L	0.5 mL of 1000 ug/L Se	100 mL
10.0 ug/L	1 mL of 1000 ug/L Se	100 mL
20.0 ug/L	2 mL of 1000 ug/L Se	100 mL
50.0 ug/L	5 mL of 1000 ug/L Se	100 mL

3. **Nickel Nitrate (0.25%):** In a 100 mL volumetric flask dissolve 1.25 g of $Ni(NO_3)_2 \cdot 6H_2O$ in D.I. water and dilute to 100 mL. Prepare fresh every 6 months.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Chloride (> 800 mg/L) and sulfate (> 200 mg/L) interfere with this selenium procedure. Nickel nitrate is added as a matrix modifier to minimize these interferences.

3. Background correction is required.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

For concentration mode, use the 5.0, 10.0, 20.0 and 50.0 mg/L standards for instrument calibration and follow the procedure for analyzing using the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   - If the spike recovery is within 85 - 115%, standard additions are not required.

   - If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference standard will be analyzed with each analysis.

Calculations:

1. Calculate using the instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-279]

Effective: 7-2-91

SILVER - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 272.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.0005 mg/L

Optimum Range: 0.0005 - 0.010 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Height
Lamp Position:	7
Lamp Current (mA):	4
Slit Width (nm):	0.5
Slit Height:	Normal
Wavelength (nm):	328.1
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	1.30

FURNACE PARAMETERS

Step	Temp (*C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	85	5.0	3.0	NORMAL	NO
2	95	40.0	3.0	NORMAL	NO
3	120	10.0	3.0	NORMAL	NO
4	400	5.0	3.0	NORMAL	NO
5	400	1.0	3.0	NORMAL	NO
6	400	2.0	0.0	NORMAL	NO
7	2000	0.9	0.0	NORMAL	YES
8	2000	2.0	0.0	NORMAL	YES
9	2000	2.0	3.0	NORMAL	NO

Graphite Tube Type: Pyrolytic coated partition tube

Sample Volume: 20 uL

Standards to use for curve set-up: 1.00, 4.00, 10.0 ug/L

Sample Handling: Acidify with nitric acid to pH < 2. Analyze within 6 months.

Reagent Preparation:

1. **Standard Silver Solution (1000 ug/L Silver):** Pipet 1.00 mL of the 1000 ppm stock silver solution into a 1000 mL volumetric flask, add 5.0 mL HNO_3 and dilute to the mark with D.I. Prepare fresh daily.

2. **Working Silver Standard (100 ug/L Silver):** Pipet 10 mL of the 1000 ug/L silver standard into a 100 mL volumetric flask and dilute to the mark with D.I. Prepare fresh daily.

3. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Silver Standard	Dilute to
1.00 ug/L	1 mL of 100 ug/L Ag	100 mL
4.00 ug/L	4 mL of 100 ug/L Ag	100 mL
10.0 ug/L	10 mL of 100 ug/L Ag	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. Background correction is required.

4. The use of halide acids should be avoided.

5. Silver standards are light sensitive and tend to plate out on the container walls. Silver standards should be stored in amber glass bottles rather than plastic.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

For concentration mode, use the 1.0, 4.0 and 10.0 standards and follow the procedure for analyzing using the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within acceptable ranges (90-110% of the true value) or the samples run after the last acceptable calibration standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. **Criteria for standard additions are:**

   - If the spike recovery is within 85 - 115%, standard additions are not required.
   - If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference standard will be analyzed with each analysis.

Calculations:

1. Calculate using instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-277]

Effective: 7-2-91

SODIUM - VARIAN 20

Method: Flame Emission: Direct Aspiration

Reference: "Standard Methods for the Examination of Water and Wastewater", 16th Edition, Method 325B, 1985

"Analytical Methods for Flame Spectrophotometry", Varian, 1979

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 1.0 mg/L

Optimum Range: 1.0 - 100 mg/L

Sample Handling: Acidify with nitric acid to pH <2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

1. Set signal to emission. (No lamp is required.)
2. Wavelength: 589.0 nm
3. Slit Width: 0.2 Normal
4. Fuel: Acetylene
5. Oxidant: Air
6. Type of flame: Oxidizing, lean, blue
7. Standards to use for curve set-up: 1.0, 5.0, 10.0, 25.0, 50.0, 75.0, 100.0 mg/L.

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard Sodium Solution (100 mg/L Sodium):** Pipet 10 mL of the 1000 ppm stock sodium solution into a 100 mL volumetric flask, add 1/2 mL HNO_3, and dilute to the mark with D.I. water.

2. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Sodium Standard	Dilute to
1.0 mg/L	1 mL of 100 mg/L Na	100 mL
5.0 mg/L	5 mL of 100 mg/L Na	100 mL
10.0 mg/L	1 mL of 1000 mg/L Na	100 mL
25.0 mg/L	2.5 mL of 1000 mg/L Na	100 mL
50.0 mg/L	5 mL of 1000 mg/L Na	100 mL
75.0 mg/L	7.5 mL of 1000 mg/L Na	100 mL
100.0 mg/L	10 mL of 1000 mg/L Na	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Flame - Direct Aspiration section of this manual but make the following changes:

1. Turn the burner head counter clockwise as far as it will go (approximately a 45° angle).

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The emission readings should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, flame head alignment, etc.).

2. A quality control calibration standard of 25.0 mg/L and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

4. An EPA reference sample will be analyzed with each analysis.

Calculations:

1. Calculate using instrument concentration mode, or

2. For method of standard additions, use linear regression.

[rff-metcont-276]

Effective: 1-14-92

THALLIUM - 400 VARIAN

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 279.2

"Analytical Methods for Zeeman Graphic Tube Atomizers" - Varian 1986.

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	10
Slit Width (nm):	0.5
Slit Height:	Normal
Wavelength (nm):	276.8
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	0.55

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	240	40.0	3.0	NORMAL	NO
3	240	10.0	3.0	NORMAL	NO
4	500	5.0	3.0	NORMAL	NO
5	500	10.0	3.0	NORMAL	NO
6	500	1.0	0.0	NORMAL	NO
7	2400	1.0	0.0	NORMAL	YES
8	2400	2.0	0.0	NORMAL	YES
9	2400	1.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix Modifier Volume: 5 uL (1% H_2SO_4)

Calibration standards: 10.00, 25.00, 50.00 ug/L.

Graphite Tube Type: Pyrolytic coated plateau tube

Sample Handling: Acidify with nitric acid to pH <2. Analyze within 6 months.

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard Thallium Solution (1000 ug/L Thallium):** Pipet 1.00 mL of the 1000 ppm stock thallium solution into a 1000 mL volumetric flask, add 0.5 mL HNO_3 and dilute to volume with D.I. water. Prepare fresh every month.

2. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Thallium Standard	Dilute to
10.0 ug/L	1.0 mL of 1000 ug/L Tl	100 mL
25.0 ug/L	2.5 mL of 1000 ug/L Tl	100 mL
50.0 ug/L	5.0 mL of 1000 ug/L Tl	100 mL

3. H_2SO_4 (1%): Add 1.0 mL of concentrated H_2SO_4 to 90 mL D.I. water.Dilute to 100 mL.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. 1% H_2SO_4 is added as a matrix modifier.

4. The use of background correction is required.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

For the use of the concentration mode, use the 10.0, 25.0 and 50.0 mg/L standards for instrument calibration and follow the procedure for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. **Criteria for standard additions are:**

   - If the spike recovery is within 85 - 115%, standard additions are not required.

   - If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference standard will be analyzed with each analysis.

Calculations:

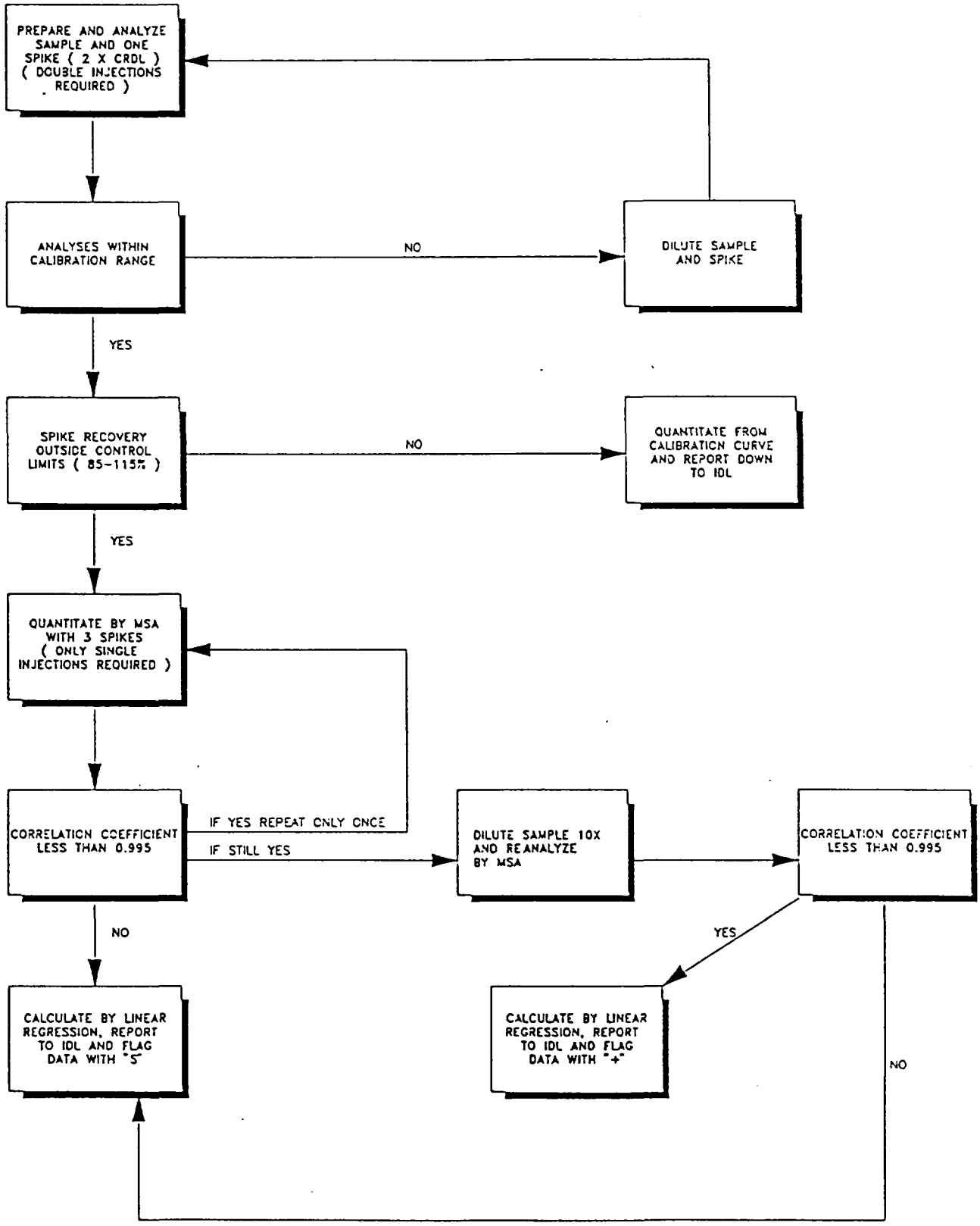
1. Calculate using instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-274]

WARZYN

FURNACE CLP DECISION TREE



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Effective: 7-2-91

VANADIUM-VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 286.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	10
Slit Width (nm):	0.2
Slit Height:	Normal
Wavelength (nm):	318.5
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	1.80

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	95	5.0	3.0	NORMAL	NO
2	130	40.0	3.0	NORMAL	NO
3	1400	10.0	3.0	NORMAL	NO
4	1400	10.0	3.0	NORMAL	NO
5	1400	1.0	0.0	NORMAL	NO
6	2700	0.7	0.0	NORMAL	YES
7	2700	2.0	0.0	NORMAL	YES
8	2700	2.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Standards to use for curve set-up: 10.0, 20.0, 50.0 ug/L.

Graphite Tube Type: Pyrolytic coated partition tube

Sample Handling: Acidify with nitric acid to pH < 2. Analyze within 6 months.

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard vanadium solution (1000 ug/L vanadium):** Pipet 1.0 mL of the 1000 ppm stock vanadium solution into a 1000 mL volumetric flask, add 1/2 mL HNO_3 and dilute to the mark with deionized water. Prepare fresh daily.

2. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Vanadium Standard	Dilute to
10.0 ug/L	1.0 mL of 1000 ug/L V	100 mL
20.0 ug/L	2.0 mL of 1000 ug/L V	100 mL
50.0 ug/L	5.0 mL of 1000 ug/L V	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of background correction is required.

4. The use of halide acids should be avoided.

5. Vanadium is a refactory metal, extra care should be taken that sample is not boiled during the digestion (vanadium is easily lost).

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Aspiration section of this manual.

For the use of concentration mode, use the 10.0, 20.0 and 50.0 standards and follow the procedure for using the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, flame head alignment, lamp alignment, etc.)

2. A quality control calibration standard of 20.0 ug/L and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. **Criteria for standard additions are:**

   - If the spike recovery is within 85 - 115%, standard additions are not required.

   - If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference sample will be analyzed with each analysis.

Calculations:

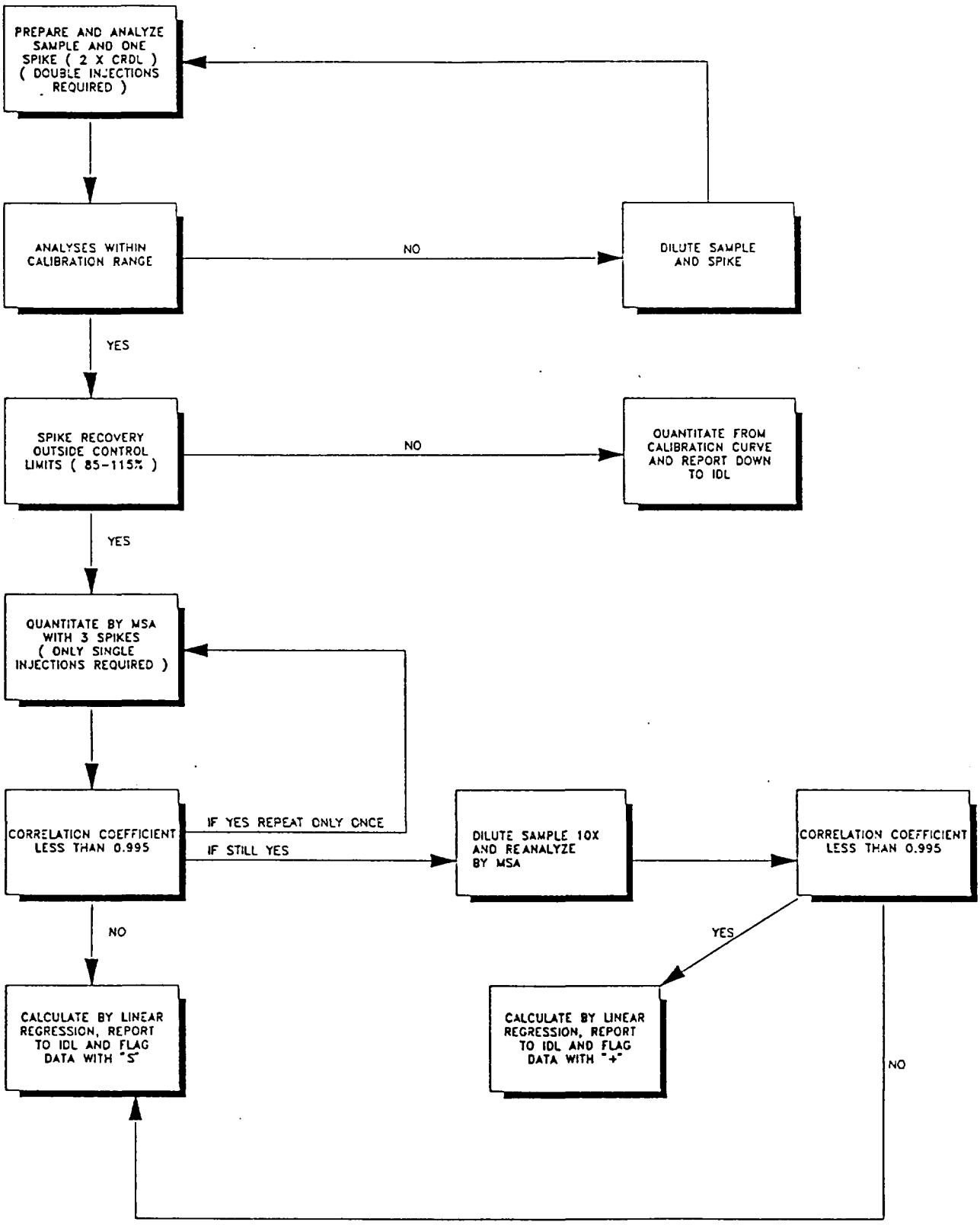
1. Calculate using instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-272]

WARZYN

FURNACE CLP DECISION TREE



Effective Date: 5.24.91

CYANIDE, TOTAL - DISTILLATION

Scope and Application: This method is applicable to the determination of cyanide in drinking water, surface water, ground-water, sludges, soils and industrial wastes.

Methods: Distillation, Automated Colorimetric

Reference: EPA 1983, Method 335.2,
SW-846, Method 9012
Standard Methods, 16th Edition, Method 412

Detection Limit: 0.005 mg/L

Optimum Range: 0.005 - 0.400 mg/L

Sample Handling: Preserve with sodium hydroxide to pH > 12 and refrigerate at 4°C
Analyze samples within 12 days of receipt date.

Reagents and Apparatus:

1. Cyanide reflux distillation apparatus
2. 25 mL and 50 mL graduated cylinders
3. Vacuum pump
4. Heating mantle
5. 250 mL volumetric flasks
6. Sodium hydroxide
7. Sulfuric acid, concentrated
8. Magnesium chloride
9. Deionized water
10. Bismuth nitrate
11. Sulfamic acid
12. Acetic acid, concentrated
13. Sodium thiosulfate, crystals

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. **Sodium hydroxide (1.25N):** Dissolve 50.0 g NaOH in D.I. water and dilute to 1 liter in a volumetric flask. Store in a plastic bottle.

2. **Magnesium chloride solution:** Dissolve 510.0 g $MgCl_2 \cdot 6H_2O$ in D.I. water and dilute to 1 liter. Store in a plastic bottle.

3. **Stock cyanide solution (1000 mg/L):** Dissolve 0.6275 g KCN and 0.5 g KOH and dilute to 250 mls with D.I. water in a volumetric flask. Prepare fresh every month. **Caution: Toxic!** Refrigerate.

4. **Standard cyanide solution (5 mg/L):** Pipet 5 mL of stock cyanide solution into 1 L volumetric flask containing approximately 500 mL D.I. water and 2 mL of 10N NaOH as a preservative. Dilute to volume with DI water. **Prepare fresh daily.** Refrigerate.

5. **Bismuth nitrate solution:** Dissolve 30.0 g of $Bi(NO_3)_3$ in 100 mL of D.I. water. While stirring, add 250 mL of concentrated acetic acid. Stir until dissolved. Dilute to 1 liter with D.I. water.

6. **Sulfamic acid solution:** Dissolve 40.0 g of sulfamic acid in D.I. water. Dilute to 1 liter.

Notes:

1. **Caution:** Use care in handling of samples with cyanide because of the toxicity. Avoid skin contact, inhalation, or ingestion. **Always have a respirator on when doing this test.**

   If a sample begins to bump or back up the tube, quickly increase the flow rate, and turn the heat down (or off) until bumping subsides.

   If a sample does boil over, proceed as follows:

   - Pull inlet tube out
   - Turn heat off (For your protection, use gloves.)
   - Put sample and heating mantle into hood
   - When sample is cool remove from mantle and heat mantle in hood on high until acid fumes have dissipated.

2. Oxidizing agents, such as chlorine, interfere by decomposing cyanides. If chlorine is believed to present, put a drop of sample on potassium iodide starch paper. If paper turns bluish, add a few crystals of sodium thiosulfate ($Na_2S_2O_3$) to the sample, mix, and retest. Continue adding sodium thiosulfate until free from chlorine. Then, add 0.1 g sodium thiosulfate in excess.

3. Sulfides interfere by forming thiocyanate at a high pH. If sulfides are believed to be present, put a drop of sample on lead acetate test paper treated with acetic acid buffer solution at ph4. Darkening of paper indicates sulfides. If sulfides are present, add 50 mL of bismuth nitrate solution after the air rate is set through the air inlet tube. Mix for 3 minutes prior to addition of H_2SO_4.

   Alternatively, $Cd(NO_3)_2 \cdot 4H_20$, $CdCO_3$ or $PbCO_3$ can be added after the distillation, but prior to color development. Bismuth nitrate added prior to the distillation process is the preferred choice.

4. Fatty acids, high carbonates, and aldehydes can interfere. Refer to Standard Methods for troubleshooting.

5. High concentrations of NO_3 and NO_2 can give false positive results. If samples contain high concentrations of NO_3 and/or NO_2, add 50 mL of sulfamic acid solution after the air rate is set through the air inlet tube. Mix for 3 minutes prior to addition of H_2SO_4.

6. Do not use bismuth nitrate and sulfamic acid aon the same sapmle. Pretreatment with both results in poor (bias low) cyanide recovery.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed, and deionized rinsed prior to analyses. Dichromate or acetone may also be used to clean the glassware prior to the soap and water wash.

2. Connect and set up cyanide reflux distillation apparatus as shown in Figure 2.

3. Prepare the 0.100 mg/L cyanide digestion standard as follows:

   Add 5 mL of the 5 mg/L cyanide solution to 250 mL of DI water. (Prepare in the distillation flask.)

4. Pour 250 mL of sample into cyanide distilling flask. If a solid or semi-solid sample is to be analyzed, use a 1.0 g sample size and add 250 mL of D.I. water to the distilling flask. (Record the amount of sample used.) Add an additional 250 mL D.I. water for a total volume of 500 mL in the distillation flask. Add 3-5 boiling chips.

   To Spike: Add 5 mL of the 5 mg/L cyanide solution to the 250 mL of sample for a final concentration of 0.100 mg/L.

5. Using a graduated cylinder, add 50 mL 1.25 N sodium hydroxide to the absorber tube and connect.

6. Turn on vacuum pump and adjust so that one bubble per second enters the distillation flask through the air inlet tube.

7. Slowly add 25 mL concentrated sulfuric acid through the air inlet tube. Rinse the tube with D.I. water and wait for about 2-3 minutes, until the sulfuric acid has been dispersed into the sample.

8. Using a graduated cylinder, add 20 mL magnesium chloride solution into the air inlet tube and rinse the tube with D.I. water.

9. Turn heating mantle on to setting of 10 till sample boils (approximatley 15 minutes). Watch vacuum rate carefully and adjust as necessary maintaining a rate of one bubble per second. As temperature increases, bubbling increases, and the solution can be drawn from the absorption tube or blown out the air inlet tube.

10. After sample starts boiling, turn heating mantle down to 6-7. **Watch vacuum rate carefully and adjust as necessary** maintaining a rate of one bubble per second. Reflux for one hour.

11. Turn off heat and continue vacuum for 15 minutes.

12. Remove inlet tubes.

13. Disconnect absorber, DI rinse absorber top into absorbing solution, and shut off vacuum pump.

14. Pour solution from absorber tube into a 250 mL volumetric flask. Using D.I. water, rinse the absorption tube (3 times) and add to the volumetric flask. Dilute to mark with DI water. Mix by inverting.

15. Distillates are ready for analysis. Proceed with Lachat SOP CNAAHC for the automated colorimetric step.

Quality Control:

1. The standard curve does not need to be carried through the distillation procedure.

2. A reagent blank is to be analyzed with each set of samples. This blank is to be carried through the distillation steps as a check for contamination.

3. A quality control distilled check standard of 0.100 mg/L cyanide is to be analyzed with each set of samples. This standard is to be carried through the entire procedure including the distillation step.

4. A known reference standard (LCS) is to be analyzed with each set of samples. This standard is to be carried through the entire procedure including the distillation steps. This standard must be within 80-120% of the true value and within 95% confidence limits (if available) or the samples are to be reanalyzed.

5. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges.

6. Aqueous and solid/semi-solid samples are separate matrices. Duplicates and spikes are required for each matrix type.

Calculation:

1. Calculate distillate concentration with Lachat QuikChem software, in the concentration mode, using the IBM XT computer. (Be sure to calculate in any distillation dilution into the final result.)

$$\text{ug/L CN} = \frac{(\text{distillate volume,mL})(\text{distillate concentration,mg/L}) \times 1000}{(\text{sample volume,mL})}$$

$$\text{mg/kg CN} = \frac{(\text{distillate volume,mL})(\text{distillate concentration,mg/L})}{(\text{sample weight,gm})}$$

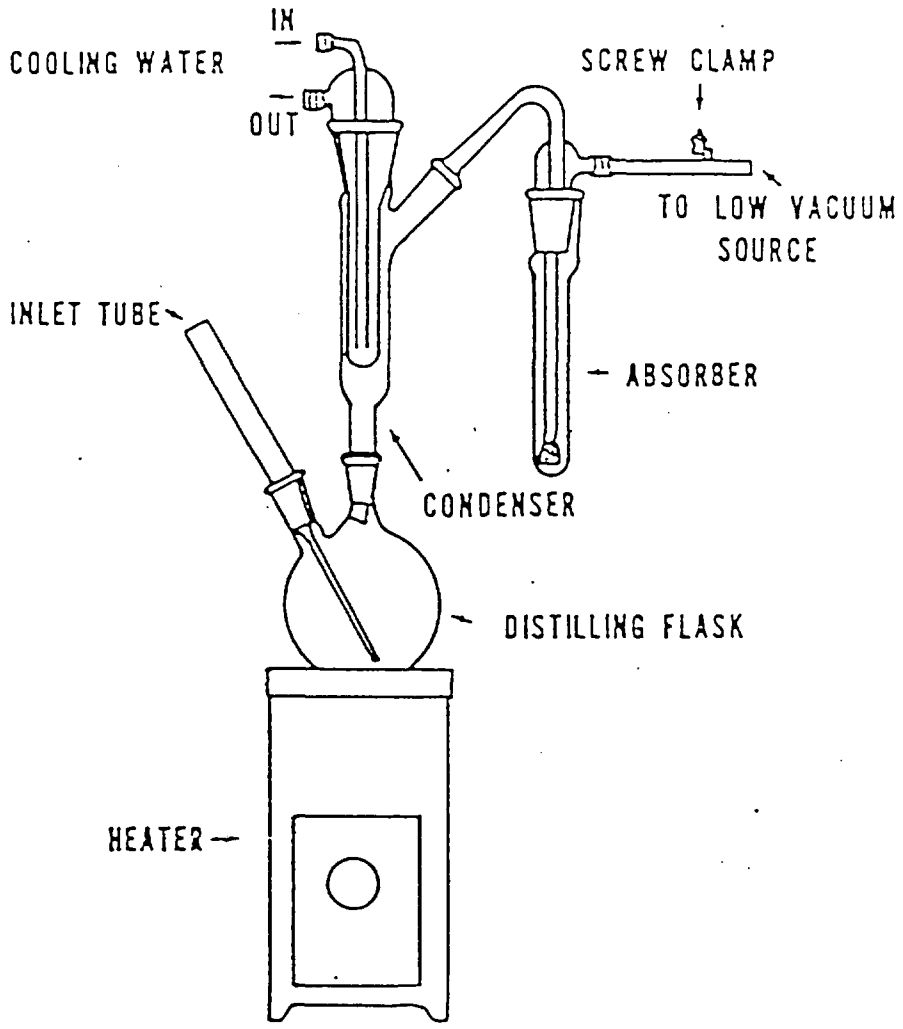


FIGURE 2

CYANIDE DISTILLATION APPARATUS

Effective Date: 5/24/91

TOTAL CYANIDE - AUTOANALYZER - (HEATED METHOD)

Scope and Application: This method is applicable to **distilled** groundwater, drinking water, wastewater, sediments and soils. All samples must be distilled prior to analysis with the autoanalyzer. (Refer to SOP # CNDISC.)

Reference: EPA, 1983, Method 335.3
Lachat Instruments, 1990, Method 10-204-00-1-A
Standard Methods, 16th Edition, pages 337-338
SW-846, Method 9012
SOW No. 788 including Rev 2/89 and 6/89

Instrument Detection Limit: 0.010 mg/L

Optimum Range: 0.005 - 0.400 mg/L

Sample Handling: Samples should be capped and refrigerated at 4°C after distillation. Samples must be analyzed within 3 days after distillation and within 12 days of receipt date.

Instrument Conditions:

1. Pump speed: 35
2. Cycle period: 50 seconds
3. Load period: 20 seconds
4. Inject period: 15 seconds
5. Inject to start of peak period: 30 seconds
6. Inject to end of peak period: 78 seconds
7. Gain: 420
8. Zero: 350
9. Interference filter: 570 mm
10. Sample loop: 150 cm (0.80 mm i.d.)
11. Standards for calibration: 0, 0.010, 0.020, 0.100, 0.200, 0.400 mg/L
12. Water Bath 45°C (Position A).

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Degassed Milli-Q-water - 2 options:**

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.

   b. Bubble helium, using the fritted gas dispersion tube, through 20 L Milli-Q water for 15-20 minutes. Store in cubitainer.

2. **Carrier - 0.25N NaOH:** In a 1 L volumetric flask, dissolve 10.0 g NaOH in 900 mL DI water. Dilute to the mark and invert several times. Filter through 0.45 micron filter paper. Store in a plastic bottle.

3. **Phosphate buffer - 0.71 M:** In a 1 L volumetric flask, dissolve 97.0 g anhydrous potassium dihydrogen phosphate (potassium phosphate, monobasic, anhydrous, KH_2PO_4) in 800 mL degassed MQ water. Add 8.1 mL concentrated (85%) phosphoric acid. Dilute to the mark with degassed MQ water and invert several times.

4. **Chloramine-T solution:** In a 500 ml volumetric dissolve 2.0 g of chloramine-T in degassed Milli-Q. Dilute to the mark and invert several times. Prepare fresh weekly and store refrigerated.

5. **Pyridine - barbituric acid reagent:** In the fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100 mL of degassed MQ water, rinsing down the sides of the beaker to wet the barbituric acid. Add 75 mL pyridine (C_5H_5N) while stirring with a stir bar. Mix until barbituric acid dissolves. Add 15 mL concentrated HCl and stir. Transfer to a 1 L volumetric flask, dilute to the mark with degassed MQ water and invert several times. Refrigerate. Prepare fresh every 2 months.

6. **Stock cyanide solution (1000 mg/L):** Dissolve 0.6275 g KCN and 0.5 g KOH and dilute to 250 mL with D.I. water in a volumetric flask. Prepare fresh every month. Refrigerate. **Caution: Toxic!**

7. **Standard cyanide solution (5.0 mg/L):** Pipet 5 mL of stock cyanide solution into 1 L volumetric flask, add approximately 500 mL DI water. Add 2 mL of 10N NaOH as a preservative and dilute to volume with DI water. Prepare fresh daily. Refrigerate.

8. **Cyanide standards:** Prepare by pipetting the volumes noted below into 250 mL volumetric flasks, adding 50 mL of 1.25N NaOH, and diluting to the mark with degassed MQ water. (The 1.25N NaOH must be added - very important!) **Prepare fresh daily.**

Concentration of Standard	Letter Identifier	Volume of 5.0 mg/L working standard (ml)	Dilute to
0.000 mg/L	A	0 mL	250 mL
0.010 mg/L	B	0.5 mL	250 mL
0.020 mg/L	C	1.0 mL	250 mL
0.100 mg/L	D	5.0 mL	250 mL
0.200 mg/L	E	10 mL	250 mL
0.400 mg/L	F	20 mL	250 mL

Note: Computer refers to standards by letter.

Notes:

1. This chemistry is temperature sensitive. The heated method reduces or eliminates sensitivity drift due to temperature changes.

2. Use wasteline coil to help eliminate air spikes.

3. Any sample dilutions must be diluted with 0.25N NaOH, **not** water. You may use the carrier or the zero standard for this.

4. Interferences are reduced or eliminated by the distillation procedure. Cyanide analyses suffer from many interferences. See EPA and Standard Methods references for detailed discussion. Information and recommendations for the manual pyridine-barbituric acid color development also apply to this automated method.

5. Samples must be diluted to obtain concentrations within the optimum working range.

6. The gain and zero settings are guidelines and must be optimized each day.

7. Color is an interference, dilute the sample and also manually spike the dilution to confirm the quality of the result.

System Operation:

1. Refer to "Auto Analyzer Operation Start-up Procedure" (IOP# LAAC-Section A).

2. Analyze an initial calibration check standard, a blank, a distilled known reference standard, a distilled standard and a distilled blank at the beginning of each run. The blanks must be below the detection limit and the standards must be within required control limits before any samples are analyzed.

3. Spikes are be distilled at a level of 0.100 mg/L.

4. The calibration check standard is 0.100 mg/L (D).

5. The distilled standard is 0.100 mg/L.

6. If a sample and spike are **overrange:**

   a. Dilute the sample and spike if dilution ≤ 1:5. The distilled spike should be detectable.
   b. Dilute the sample, spike and analyze a manual spike if dilution > 1:5.

7. Refer to Auto Analyzer shut-down procedure. (IOP# LAAC-Section B).

Quality Control:

1. Establish a standard curve with the standards listed above. The derived concentrations for each calibration standard must read within 10% of the true value. The derived value for the blank must be less than the method detection limit.

2. A quality control calibration check standard of 0.100 mg/L (D) and a blank are to be analyzed initially and at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration check standard and blank are still required. The last samples analyzed in the run are to be the calibration check standard and blank. These standards must be within the acceptable ranges and blanks must be below the method detection limit or the samples run after the last acceptable calibration check standard and blank are to be reanalyzed.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicates are to be within acceptable ranges or data must be flagged appropriately. (These samples must be carried through the distillation step.)

Calculations:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer. Be sure to calculate any digestion dilution into the final result.

APPENDIX B

GENERAL STANDARD OPERATING PROCEDURE FOR SKINNER AND SHERMAN LABORATORY

INTERNAL QUALITY CONTROL

13.1 PERFORMANCE CHECKS OF RADIATION SCREENING EQUIPMENT

Performance checks are made to assure the continuing capability of the screening instruments. Procedures include efficiency checks, and background determinations. The procedure and frequency of each check is optimized for each detector system to provide assurance of the detector's performance. Documentation of the checks and the results are kept for all procedures. The Radiation Safety Officer is responsible for all performance checks. Detailed procedures and schedules are outlined in the "Radiation Protection Plan" of the NRC license application.

13.2 ENVIRONMENTAL LABORATORY SERVICES

13.2.1 INTERNAL QUALITY CONTROL CHECKS

The Supervisors or Program Data Managers are responsible to ensure that analytical results are produced internally within acceptable limits.

13.2.1.1 PRECISION

Replicate standards and/or samples are used to estimate the precision of each analytical test procedure for a known matrix. Data control limits are established to satisfy the requirements of specific measurement projects, based on prior knowledge of the measurement system and method validation studies or state certification limits.

13.2.1.2 ACCURACY

Certified standards and/or spiked samples are used to estimate analyte recovery for each test procedure for a known matrix.

13.2.1.3 DUPLICATE AND SPIKES

Duplicate sample and/or matrix spike and matrix spike duplicate samples are analyzed with each set of samples, every 20 samples, 5 percent, or every 10 samples, whichever is appropriate.

13.2.1.4 COMPLETENESS

Data control limits are based on the mean precision and accuracy values determined in the laboratories or based on EPA Contract Laboratory Protocol requirements. When practical, acceptance limits are set at the mean values plus or minus three standard deviations. Samples outside the control limits are reanalyzed during a successive testing period or flagged where appropriate.

13.2.1.5 REPRESENTATIVENESS

Whenever possible, samples are collected and aliquotted so that the analysis results are representative of the media and conditions being measured.

13.2.1.6 COMPARABILITY

Unless otherwise specified, all data are calculated and reported in units consistent with other organizations reporting similar data to allow comparison of data bases.

Table 13.1 PERFORMANCE OBJECTIVES FOR GENERAL QC PARAMETERS

QC PARAMETER	PERFORMANCE OBJECTIVES
Laboratory and Trip Blanks. Detect contamination during shipping, handling and preparation.	Results less than contract required detection limit. Once per batch per matrix and concentration level (high, medium, low).
Initial and Continuing Calibration Blanks. Determine background levels.	Results less than contract or client required detection limit. Every 10 samples for inorganics and wet chemistries or every 12 hours for organics.
Spike Samples and Duplicate Samples. Samples spiked during preparation that determine accuracy, precision and detect matrix effects.	If unspecified, recoveries should be 75-125% of the theoretical value. Once per batch per matrix per concentration level.
Duplicate Samples. Preparation and analysis performed to determine precision.	Relative percent difference should be less than 20% unless otherwise specified. Once per batch per matrix per concentration level.
Initial Calibration Verifications. Independent source standard analyzed to validate instrument calibration.	Recoveries between 90-110%. May also be analyzed at the end of sample analysis run.
Continuing Calibration Verifications. Confirm the calibration throughout the sample analyses.	Recoveries between 90-110%. If unspecified, every 10 samples for inorganic and wet chemistries, and every 12 hours for organics.
Surrogate Standards and Internal Standards. Estimate recoveries and account for sample variations and matrix effects.	As specified by contract, client or Standard Operating Procedure.
External/Blind Performance Evaluation Samples. Assure laboratory wide compliance with QC objectives.	Recoveries within published acceptance levels. Performed twice per test method per year.
Laboratory Control Standards and Method Spikes. Determine method interferences and accuracy.	Recoveries between 75-125% of theoretical. Once per batch unless otherwise specified.

13.2.3 SAMPLE CUSTODY

All samples are assigned a unique internal laboratory identification number, marked directly on the container to denote customer number and sample number. The laboratory shall designate a Sample Custodian and a back-up Sample Custodian for chain of custody samples. Lockable refrigerators, freezers, and cabinets are available for controlled samples. Internal sample custody forms are used for tracking all these samples through the analytical process.

13.2.4 STANDARD PREPARATIONS

All standards are prepared from certified solutions or reagent materials that are checked against certified standards. A complete record, including preparation date, individual preparing solutions, and the person who prepared the standard is maintained in a standards preparation logbook. Laboratory personnel are instructed to label standard's container.

13.2.5 COLLABORATIVE TESTING

In addition to the internal QC program, the laboratory participates in collaborative testing or interlaboratory comparison programs. This includes participation in numerous round robin, interlaboratory studies, and Quality Control testing programs. At a minimum, the laboratory actively participates in the U.S. EPA InterLaboratory Quality Control Program for Water Supply/Water Pollution.

13.3 DATA VERIFICATION

Data reliability is based on method specific quality control limits worked into data processing systems at the bench level. Calculated precision and accuracy control limits are used to flag outliers. Overall data compatibility is performed by checking for proper reporting units and minimum and maximum reporting ranges. The laboratory Supervisor reviews and initials the input data. All raw data, including the final results, are stored for later review. Calculations performed manually are reviewed by a second person and initialed as checked.

13.3.1 ELECTRONIC DELIVERABLES VERIFICATION

Supervisors and/or Program Managers are responsible for assuring that electronic deliverables accurately reflect the hardcopy deliverables.

13.4 DATA SECURITY

Computer Software Systems used in Data Handling and information and reporting use password-based security levels for all who have access to the computer network and data base. Data and information is archived on a daily and weekly basis.

13.5 TECHNICAL COMPLAINTS

Technical Complaints are addressed by the Program Manager or staff member with the most expertise in the field of complaint with support from the Technical Director. Upon receipt of the technical complaint, the Program Manager assigns the data inquiry a number and initiates a Data Inquiry Form. The form will contain the work order number, client, and concern. The Supervisor will receive this form attached to the work order file for review. The Supervisor will then determine if the complaint is valid, fill out the Data Inquiry Form and return it to the Program Manager for client call back. If the complaint is determined to be valid, then the cause for the complaint will be corrected as soon as feasible. A copy of the Data Inquiry Form will be stored in the case file. The original will be maintained by the QA Officer.

PREVENTIVE MAINTENANCE AND INSTRUMENTATION REDUNDANCY

9.1 POLICY

Preventive maintenance is performed as required on analytical instrumentation to prevent down time of the measuring systems.

9.2 MAINTENANCE

Maintenance procedures are developed by the Supervisors for use where procedures are not provided in the manufacturer supplied operator's manual. Records of instrument set-up, maintenance, and repair are maintained. The Supervisors and operating personnel are responsible for complying with the instrument maintenance schedule.

9.3 SPARE PARTS

Supervisors and operators are responsible to assure that an adequate inventory of spare parts and consumables are requisitioned and maintained for the instrumentation in their area(s) in order to prevent downtime or compromised running conditions.

9.4 INSTRUMENTATION REDUNDANCY

Back-up instrumentation, autosamplers, and computer software systems used to perform routine processes are maintained where necessary.

Redundancy exists for the following instrumentation and equipment: Laboratory Information Management System (LIMS) Database and hardware, LIMS Network Fileserver and Hardware, Gas Chromatography-Mass Spectrometry, Gas Chromatography, High Performance Liquid Chromatography, Wet Chemistry Autoanalyzer, Inductively Coupled Argon Plasma Spectrophotometer, Graphite Furnace Atomic Absorption Spectrophotometer, Cold-Vapour Mercury Analyzer, Ion Selective Electrodes, Refrigerators, Ovens, and Distillation, Digestion and Extraction equipment.

CORRECTIVE ACTION

16.1 POLICY

In order to maintain and assure acceptable quality levels for services provided by TMA/Skinner & Sherman, a feedback system shall be established to assure that conditions adverse to quality shall be promptly identified and corrected. Corrective actions shall be determined and initiated as indicated below.

16.2 CORRECTIVE ACTION

16.2.1 The QA Officer shall initiate investigation and corrective action by using a Corrective Action Request Form in any of the following situations:

16.2.1.1 When an audit reveals circumstances that may adversely affect quality as determined by the QA Officer.

16.2.1.2 When the results of an intercomparison study program are out of control.

16.2.1.3 When procedural or technical problems arise and the QA Officer or supervisors determine that they may significantly affect quality.

16.3 RESPONSIBILITY

All personnel are responsible to communicate any evidence of unacceptable quality performance to the Supervisor and/or the QA Officer, or laboratory management.

16.3.1 The Supervisor or designee shall be responsible for investigating conditions adverse to quality, determining the assignable cause and recommending the actions necessary for their correction.

16.3.2 The General Manager or designee shall provide pertinent information regarding cause of adverse conditions and then determine the specific corrective action necessary to preclude recurrence.

16.3.3 The QA Officer shall review the Corrective Action trends and other routine Quality Control data for evidence of unacceptable quality.

16.3.4 All items requiring corrective action shall be clearly identified in the Corrective Action Request Form for subsequent follow-up and close-out actions. Completed copies of the Corrective Action Request Form shall be kept by the QA Officer.

"Internal Sample and Extract Custody"
TMA/Skinner & Sherman Laboratories, Inc.

Approval: Joan Ladava — Joah Ladava, General Manager
Lisa Snow — Lisa Snow, QA Officer

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1. Scope and Applicability:

This SOP describes practices employed by TMA/Skinner & Sherman concerning the procedures and the documentation of sample and extract transfers for controlled samples. The procedure starts upon transmittal of the Internal (SAM) Chain of Custody and ends with sample purge.

2. Reference:

2.1 USEPA CONTRACT LABORATORY PROGRAM STATEMENT OF WORK, Document Number ILM01.0 (SOW 3/90).

2.2 TMA/Skinner & Sherman Quality Assurance Manual

3. Procedures:

3.1 Internal Storage

3.1.1 The sample custodian checks that each Internal Chain of Custody has the appropriate storage locations (Section 3.1.1) and sample identifications.

3.1.2 The corresponding Extract, Distillate, Digestate Chain of Custodies (COC's) are delivered to or posted at the area where the samples are originally stored or where the extracts, digestates or distillates are originated by the sample custodian.

3.1.3 The raw sample Internal COC's remain at their original post until the whole batch of samples have been analyzed and are relocated by the sample custodian, then the COC is moved into a COC folder maintained in sample control with the documented move.

3.1.4 For EPA/CLP samples copies of the Internal COC's are included in the data package. The Sample Custodian copies the Internal COC's and includes the copy(s) in the case file folder.

3.1.5 When the samples are moved for disposal, this is documented on the Internal COC which is then archived in the library. Raw sample disposal is documented on the raw sample COC and the Extract, Distillate, and Digestate sample disposal is documented on their COC(s).

3.1.6 Raw Sample storage is maintained in-house for a minimum of 30 days for commercial and 60 days for CLP or CLP-type projects after reporting the data.

3.1.7 EPA/CLP organic sample splits are refrigerated for 60 days after reporting and commercial sample splits are refrigerated for at least 30 days after reporting.

3.1.8 Samples are Removed and returned in batches are documented on an internal custody form. Only one sample delivery group or batch is entered per internal custody form.

3.1.9 The analyst who signs out for the sample(s) is responsible for the custody of the sample, and is responsible for returning the sample as soon as is practical after taking the aliquot(s).

3.2 Internal Chain of Custody Locations

3.2.1 CLP, CLP-TYPE Raw Inorganics Samples - For CLP raw Inorganics samples the IP internal COC is posted on the clipboard to the right of the Large (Arctic) Walk-in and is used for the all CLP Inorganic tests.

3.2.2 CLP, CLP-TYPE Digestates and Distillates - The IC internal COC is delivered to the Inorganic Prep Lab and used for internal transfers of the AA and ICP digestates. The WC internal COC is delivered to the Cyanide Distillation Lab and is used for the movement of CLP cyanide distillates.

3.2.3 Raw Commercial Organics and Inorganics - For Commercial samples that are stored in either the (REVCO) 2-Door refrigerator or the (VOLLRATH) small Walk-in, the internal COC is the overall one that has no department code specified. It is posted to the right of the REVCO 2-Door.

3.2.4 Volatiles (VOA) Raw and Extract Samples - All VOA internal COC's are posted on the clipboard to the right of the Low-level VOA refrigerator.

3.2.5 PCB/Pesticide and Semi-Volatile (BNA) Extracts - The GC and GCMS internal chain of custodies are delivered to the Organic Prep lab to document the custody of the organic PCB/Pesticide and BNA extracts, respectively, for CLP and CLP-type protocols.

3.3 Sample and Container Integrity

Broken containers are discarded of appropriately. Samples that are received broken are brought to the attention of the client representative and the sample is transferred to a duplicate container, unless otherwise requested by the client. This is documented on the chain of custody. For EPA/CLP samples call SMO.

3.4 Internal Custody Form

The TMA/Skinner & Sherman internal custody form is the CHAIN OF CUSTODY (Fig. 1) generated in the transmittal of SAM paperwork.

When the Workorder paperwork is transmitted the following departmental CHAIN OF CUSTODY forms are posted at the appropriate stations as noted above.

Fill-in the RELEASED BY/TRANSFERRED TO/RECEIVED BY section as such:

RELEASED BY	DATE	TRANSFERRED TO	DATE	RECEIVED BY	DATE

The Sample Custodian uses the top set of lines to log the samples into the storage area or refrigerator --

The second line under RELEASED BY - the sample custodian's name (initials) with date and then the first prep chemist to remove the samples after log-in.

Samples are removed in batches with the container in which they are stored. Chemists remove return the whole bucket or box of sample for the test at hand.

"Internal Sample and Extract Custody"
TMA/Skinner & Sherman Laboratories, Inc.

Fig. 1 CHAIN OF CUSTODY

RELEASED BY	DATE	TRANSFERRED TO	DATE	RECEIVED BY	DATE
"Jeff DePaolo stored 6 soils/3 waters in WA on 3/21/91"					
JD	3/21/91	Prep Lab	3/21/91	HE	3/21/91
HE	3/21/91	Arctic Walk-in	3/21/91	JD	3/21/91
JD	3/25/91	Cyanide Lab	3/25/91	BB	3/25/91
BB	3/25/91	Arctic Walk-in	3/25/91	JD	3/25/91
JD	3/28/91	HG Lab	3/28/91	LD	3/28/91
LD	3/28/91	Arctic Walk-in	3/28/91	JD	3/28/91
JD	4/25/91	Cabinet 8	4/25/91	JD	4/25/91
JD	6/26/91	Closet	6/26/91	JD	6/26/91
JD	6/26/91	Disposal	6/26/91	RL	6/26/91

3.5 Storage System

3.5.1 The location of sample storage is typed into the space under "STORED" in the SAM log-in of each sample split. The sample custodian uses the storage abbreviations listed for the location where the samples will be stored after log-in, and during analysis.

STORAGE DESIGNATIONS

COMM - VOLLRATH WALK-IN (6'x 6 x 6')
CLP - ARCTIC WALK-IN (7'x 10'x 7.8')
2-Door - REVCO DOUBLE DOOR
VOA - EQUATHERM REFRIGERATOR #2, VOLATILES
524 - EQUATHERM REFRIGERATOR #4, LOW-LEVEL VOLATILES
ORGS#3 - WESTINGHOUSE
GC #1 - REFRIGERATOR #1 FOR GC SAMPLES
METLAB - DIGESTATES FOR TOTAL METALS
CNLAB - CYANIDE DISTILLATES
DOCK - LOADING DOCK CLOSET SHELVES

3.5.2 Once analysis is complete for Inorganics raw samples any left-over sample is transferred by the sample custodian to the Office 10 cabinets as listed below. The sample organization for these cabinets is based sample types and volumes, however samples that arrived in the same months are typically stored together.

OFFICE 10 (CABINET ROOM):

C1 - LOCKABLE CABINET
C2 - " "
C3 - " "
C4 - " "
C5 - " "
C6 - " "
C7 - " "
C8 - " "
C9 - " "
C10 - BLACK CABINET NON-ROUTINE CABINETS

APPENDIX C

GENERAL STANDARD OPERATING PROCEDURES FOR ROCKY MOUNTAIN ANALYTICAL LABORATORY

Enseco

MEMORANDUM

TO: QA Directors

FROM: Peggy Sleevi PS

DATE: July 5, 1989

SUBJECT: Revision to M-EQA-002, Rev. 2.0 (Solid Matrix)

SOP M-EQA-002, Rev 2.0, Internal QC Checks--Laboratory Performance QC, requires the use of Ottawa sand as the solid matrix for Duplicate Control Samples (DCS) and Single Control Samples (SCS). Technical problems with background levels of various metals and poor precision have been observed when using Ottawa Sand as the solid matrix in metals analyses. The EPA Solid Matrix, available from USEPA EMSL-Las Vegas, is to be substituted for Ottawa sand when the solid matrix is required for metals analyses.

We will continue to use Ottawa sand as the solid matrix for organic analyses.

A typographical error appears for the solid matrix QC category for the Oil and Grease gravimetric and IR methods. The correct QC categories for the solid matrix for these methods are O&G-G-S and O&G-IR-S, respectively.

Please inform laboratory staff of these changes and retain this memorandum as documentation of these changes to M-EQA-002, Rev. 2.0.

cc: Kathy Carlberg

Enseco Incorporated
2240 Dabney Road
Richmond, Virginia 23230
804/359-1900 Fax: 804/353-1860

STANDARD
OPERATING
PROCEDURE

Subject or Title: Internal QC Checks--Laboratory Performance QC		Page 1 of 23
SOP No.: M-EQA-002	Revision No.: 2.0	Effective Date: May 15, 1989

Supersedes: M-EQA-002, Revision No. 1, June 28, 1988

1.0 PURPOSE

1.1 To describe the essential elements of Enseco's Internal QC Program related to Laboratory Performance QC.

1.2 Laboratory Performance QC consists of internal QC check samples which are used to determine whether the laboratory is "in control" during data generation. These check samples, called Laboratory Control Samples (LCS), are generated using a standard control matrix and are used to measure laboratory performance independent of sample matrix effects.

1.3 There are three types of LCS. Duplicate Control Samples (DCS), Surrogate Control Samples (SCS), and Method Blanks.

1.4 Surrogates are added to samples and QC samples for some organic methods. The monitoring of surrogate recoveries in QC samples is an element of Laboratory Performance QC. However, the monitoring of surrogate recoveries in samples is not used to control laboratory operations and thus is not an element of Laboratory Performance QC. Surrogate recoveries in samples are used to measure the effect of the matrix or method performance and are thus an element of Matrix Specific QC (See M-EQA-003).

Prepared by: Peggy Sleevi	Date: May 15, 1989
Management Approval: Peggy Sleevi for Kathy Cardone	Date: 5-19-89
QA Approval: Peggy Sleevi	Date: 5-19-89

Enseco

MEMORANDUM

TO: QA Directors

FROM: Peggy Sleevi PS

DATE: August 2, 1989

SUBJECT: M-EQA-002 Revision 2.0, Ammendment for Oil and Grease DCS

Based on the DCS data for the Oil and Grease Gravimetric method, submitted to the corporate QA group for review, the DCS component must be changed to Paraffin Oil (Baker B356 28 9388-01) to replace the Reference Oil cited on page A4-3 of SOP No. M-EQA-0002. The paraffin oil from Baker is currently in use at Cal and East. The Reference Oil cited in method 413.2 for O&G-IR (mixture of hexadecane, isooctane and chlorobenzene) will continue to be the required spiking component for the IR method. For clarity, a replacement page for Page A4-3 is attached. The other typographical errors that occurred on this page have also been addressed along with a clarification on the DCS for the aromatic method as requested by RMAL. A complete revision of this document will be addressed within the next quarter.

Please distribute this corrected page to all employees who have a copy of this document.

cc: Kathy Carlberg

Enseco Incorporated
2240 Dabney Road
Richmond, Virginia 23230
804/359-1900 Fax: 804/353-1860

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989 x

Oil and Grease

		QC Category		Spike Level	
DCS	Method	Aqueous	Solid	Aqueous (mg/L)	Solid* (mg/Kg)
Control Mixture (1)	Aromatic	O&G-AR-A	O&G-AR-S	50	1000
Paraffin Oil	Gravimetric	O&G-G-A	O&G-G-S	50	1000
Reference Oil (2)	IR	O&G-IR-A	O&G-IR-S	5+	50+

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

(1) As specifed in LM-RMA-1044 and LM-RMA-1045
(2) As specified in Method 413.2, section 6.4.1

* Assumes 50 g sample (wet weight).

+ Spike level may be adjusted to account for variation in instrument sensitivity. Alternate spike level must be within 5 times the reporting limit.

x Revision of the DCS component for Aromatic and Gravimetric methods and correction of typographical error for solid QC categories made August 2, 1989 by P. Sleevi for immediate incorporation in SOP No. M-EQA-0002, Rev. 2.0.

Peggy Sleevi 5-2-89

2.0 POLICIES

2.1 An essential element of Quality Control is to measure laboratory performance of a method. This is done using precision and accuracy data gathered from spiking a consistent, reproducible control matrix with a prescribed list of target analytes at known prescribed concentrations. A control matrix is used so that these measurements reflect laboratory performance and not matrix effects.

2.2 Quality control data, including recovery and method blank data, must be generated with every batch of samples processed.

2.3 QC information is generated and evaluated at the bench. The prime responsibility for implementation of the QC Program lies with the analyst. The QA Department serves in an audit function and addresses special QA needs such as QA Project Plans. QC information needed for standard reports is generated, documented, and entered into LIMS by the analyst.

3.0 SAFETY ISSUES

3.1 The toxicity or carcinogenicity of each chemical used in this procedure has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible level.

3.2 A Material Safety Data Sheet (MSDS) is available for each laboratory standard and reagent chemical. The appropriate MSDS must be reviewed by the employee before handling the chemical(s).

3.3 All laboratory personnel should be thoroughly familiar with the laboratory Safety Manual before undertaking any laboratory work.

4.0 PROCEDURE

4.1 Types of Laboratory Performance QC Samples.

4.1.1 Duplicate Control Sample (DCS): A DCS consists of a standard control matrix which is spiked with a group of target compounds representative of the method analytes.

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A DCS pair is analyzed for every 20 samples processed by a method. The DCS pair is used to monitor both the precision and accuracy of the analytical method on an ongoing basis, independent of matrix effects.

4.1.2 Single Control Sample (SCS): A SCS consists of a standard, control matrix which is spiked with surrogate compounds appropriate to the method. The surrogates are spiked into the method blank, which serves as both the method blank and the SCS. In cases where no surrogate is available for a method, e.g., metals analyses, the components of the SCS are identical to the DCS components and the SCS is analyzed in addition to the method blank.

A SCS is analyzed with each batch of samples processed to ensure that QC data are generated with every sample processed by Enseco. In the case where the SCS components are identic to the DCS components, e.g. metals analyses, the SCS are analyzed with every batch of samples which does not include the DCS pair.

4.1.3 Method Blank: A method blank consists of reagents specific to the method which are taken through the entire analytical process to assess the level of contamination which exists in the analytical system and which might lead to the reporting of elevated concentrations or false positives. Method blanks for aqueous samples include, in addition to reagents, an aliquot of analyte-free water equal in volume to the sample volume required in the method SOP. A method blank is analyzed with every batch of samples processed.

4.1.4 Surrogate Spikes of Samples: Certain methods, typically organic methods, specify that surrogates must be added to each sample and QC sample processed by the method. These surrogates are added routinely as specified in Appendix A.

Surrogates in QC samples are used to measure lab performance. However, surrogate recoveries in samples are not used to determine whether the laboratory was in control during data generation. Surrogate recoveries in samples are a measure of the effect of the matrix on method performance and are therefore an element of Matrix Specific QC (see SOP No. M-E 003).

4.2 Control Limits for Laboratory QC Samples.

4.2.1 DCS. DCS are monitored for accuracy (average recovery of each analyte in the DCS pair) and precision (Relative Percent Difference (RPD)) between each analyte in the DCS pair. Control limits for accuracy are based on the average recovery of each pair of DCS generated for a QC lot as described below.

4.2.1.1 Accuracy is determined by calculating the percent recovery of each spike component in the DCS using the following formula.

$$\text{DCS (\% Recovery)} = \frac{X}{T} \times 100$$

where: X = the observed value of the measurement.
T = the "true" value

The average recovery is calculated as follows.

$$\text{Average DCS Recovery} = \frac{DCS_1 + DCS_2}{2}$$

where: DCS_1 = first DCS recovery value
DCS_2 = second DCS recovery value

The control limits for accuracy are based on the average recovery of the DCS pairs. These limits are calculated from the historical average recovery of the average DCS values $\pm$ 3 standard deviation units.

Control limits are updated every six months using the most recent nine months of DCS data. CLP limits are used until sufficient data are generated to determine laboratory limits. If the calculated limits exceed CLP limits, CLP limits are implemented.

4.2.1.2 Precision is monitored by calculating the Relative Percent Difference (RPD) of the percent recoveries of each spike component in the DCS using the following formula:

$$RPD = \frac{|DCS_1 - DCS_2|}{(DCS_1 + DCS_2)/2} \times 100$$

where: RPD = Relative Percent Difference
DCS_1 = first DCS recovery value
DCS_2 = second DCS recovery value

The control limits for precision for the DCS are calculated based on the historical RPD and range from zero (no difference between DCS results) to the average RPD plus 3 standard deviation units.

Control limits are updated every six months using the most recent nine months of DCS data. CLP limits are used until sufficient data are generated to determine laboratory limits. If the calculated limits exceed CLP limits, CLP limits are implemented.

4.2.2 SCS. SCS are monitored for accuracy (percent recovery) of the spiked analytes.

Accuracy is determined by calculating the percent recovery of each spike component in the SCS.

$$\text{Percent Recovery} = \frac{X}{T} \times 100$$

where: X = the observed value of the measurement.
T = the "true" value

The control limits for accuracy for the SCS are calculated based on the historical average recovery of all SCS values ± 3 standard deviation units.

Control limits are updated every six months using the most recent nine months of SCS data. CLP limits are used until sufficient data are generated to determine laboratory limits. If the calculated limits exceed CLP limits, CLP limits are implemented.

4.2.4 Surrogate Spikes of Samples: Enseco uses the recoveries of surrogate spikes added to method blanks (SCS) to monitor laboratory performance. Surrogate recoveries in samples are not used to make decisions on whether the laboratory was "in control" during data generation; sample surrogate recoveries are a measure of the effect of the matrix on the method performance and are, therefore, an element of Matrix Specific QC (see SOP No. M-EQA-003).

4.3 Generation of Laboratory QC Samples.

4.3.1 Volatile Organics (VOA):

4.3.1.1 DCS. A pair of DCS are analyzed for every 20 samples. DCS components and spike concentrations for VOA methods are given in Appendix A, Part 1.

For aqueous samples, the DCS components are spiked into organic-free water. For solid samples, organic-free Ottawa sand is used as the matrix.

4.3.1.2 SCS. A SCS is analyzed with every analytical batch. An analytical batch for VOA's is defined as VOA's analyzed within a 12-hour period. SCS components and spike concentrations are given in Appendix A, Part 1.

For aqueous samples, the SCS components are spiked into organic-free water. For solid samples, the SCS components are spiked into the extraction solvent when the method blank and the SCS are incorporated as one sample. For methods that do not have surrogates, the SCS, analyzed in addition to the method blank, is spiked with the components in the DCS. When the SCS and method blank are analyzed as separate samples, the SCS will incorporate Ottawa sand as the solid matrix.

4.3.1.3 Method Blank. For aqueous samples, an aliquot of organic-free water serves as the method blank for VOA analyses. For solid samples, a reagent blank is used as the method blank. Ottawa sand is never incorporated into the method blank.

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4.2.3 Method Blanks. Control limits for method blanks are as follows:

4.2.3.1 For organic analyses, the concentration of target analytes in the method blank must be below the reporting limit for that analyte in order for the method blank to be considered acceptable. An exception is made for common laboratory contaminants [methylene chloride, acetone, 2-butanone, toluene and bis(2-ethylhexyl)phthalate] which may be present in the method blank at up to 5 times the reporting limit and still be considered acceptable.

4.2.3.2 For metals analyses, the concentration of the target analytes in the method blank must be below two times the reporting limit. If the method blank value for a target analyte lies below the reporting limit, the reporting limit for that analyte in the associated samples is unaffected. If the method blank value lies between the reporting limit and two times the reporting limit, the reporting limit for that analyte in the associated samples is raised to the level found in the method blank. A method blank containing analyte(s) above two times the reporting limit is considered unacceptable unless the lowest concentration of the analyte in the associated samples is at least ten times the method blank concentration (as per CLP protocol).

4.2.3.3 For conventional inorganic tests, the method SOP directs how the method blank is treated. Generally, a reagent blank is used both to zero the equipment and as one of the calibration standards. If a preparation step is required for the analysis, then a prep blank is also analyzed to determine the extent of contamination or background interference. In most cases, the concentration found in the prep blank is subtracted from the concentration found in any associated sample prior to calculating the final result. Method blanks have no application or significance for some conventional inorganic parameters (e.g. pH).

A method blank must be analyzed with every analytical batch or with every 20 samples, whichever is more frequent. An analytical batch for VOA's is defined as VOA's analyzed within a 12-hour period.

For most VOA methods, at least one designated surrogate is spiked into the method blank. These surrogates also serve as the SCS components for these methods, and therefore, the method blank and the SCS are one and the same (see Appendix A, Part 1).

4.3.1.4 Surrogates Spiked into Samples. Surrogates compounds are required to be spiked into samples and QC samples for most VOA methods. Surrogates designated by the methods and spike concentrations to use are given in Appendix A, Part 1. These surrogates should be spiked into all samples and QC samples.

4.3.2 Extractable Organics:

4.3.2.1 DCS. A pair of DCS are analyzed for every 20 samples. DCS components and spike concentrations are given in Appendix A, Part 2. For aqueous samples, DCS components are spiked into organic-free water. For solid samples, the DCS components are spiked into the organic-free Ottawa sand used as the control matrix.

4.3.2.2 SCS. A SCS is analyzed with every analytical batch. An analytical batch for extractable organics is defined as samples extracted or prepared at the same time. SCS components and spike concentrations are given in Appendix A, Part 2.

For aqueous samples, the SCS components are spiked into organic-free water. For solid samples, the SCS components are spiked into the extraction solvent when the method blank and the SCS are incorporated as one sample. For methods that do not have surrogates, the SCS, analyzed in addition to the method blank, is spiked with the components in the DCS. When the SCS and method blank are analyzed as separate samples, the SCS will incorporate Ottawa sand as the solid matrix.

4.3.2.3 Method Blank. For aqueous samples, an aliquot of organic-free water taken through the entire analytical process serves as the method blank. For solid samples, a reagent blank is used as the method blank. Ottawa sand is never incorporated into the method blank.

A method blank must be analyzed with every analytical batch or with every 20 samples, whichever is more frequent. An analytical batch for extractable organics is defined as samples extracted or prepared at the same time.

For many extractable organic methods, at least one designated surrogate is spiked into the method blank. These surrogates also serve as the SCS components. For these methods, the method blank and the SCS are one and the same (see Appendix A, Part 2).

4.3.2.4 Surrogates Spiked into Samples. Surrogate compounds are required to be spiked into samples and QC samples for many extractable organic methods. Surrogates designated by the methods and spike concentrations to use are given in Appendix A, Part 2. These surrogates should be spiked into all samples and QC samples.

4.3.3 Metals:

4.3.3.1 DCS. A pair of DCS are analyzed for every 20 samples. Separate QC Categories have been established by matrix for Dissolved, Total, and Total Recoverable Metals. DCS components and spike concentrations for metals are given in Appendix A, Part 3. For aqueous samples, the DCS components are spiked into deionized water and taken through the procedure. For solid samples, the DCS components are spiked into ~~Ottawa sand~~ EPA Solid Matrix and taken through the procedure.

4.3.3.2 SCS. The SCS has the same components as the DCS. The SCS is analyzed with every analytical batch wh does not include the DCS. An analytical batch for

metals is defined as samples which are digested at one time (total and total recoverable metals) or samples analyzed within a 24-hour period (dissolved metals). For solid samples, the SCS components are spiked into ~~Ottawa sand~~ EPA Solid Matrix.

4.3.3.3 Method Blank. For both aqueous and solid samples, an aliquot of deionized water taken through the entire analytical process serves as the method blank. Ottawa sand is never incorporated into the method blank for solid samples.

A method blank must be analyzed with every analytical batch or with every 20 samples, whichever is more frequent. An analytical batch for metals is defined as samples which are digested at one time (total and total recoverable metals) or samples analyzed within a 24-hour period (dissolved metals).

For metals analyses, the method blank is analyzed in addition to the DCS and SCS (see Appendix A, Part 3).

4.3.3.4 Surrogates Spiked into Samples. Not applicable to metals analyses.

4.3.4 Conventional Inorganics:

4.3.4.1 DCS. A pair of DCS are analyzed for every 20 samples. DCS components and spike concentrations are given in Appendix A, Part 4. For aqueous and solid samples, the DCS components are spiked into deionized water. No solid matrix is incorporated into the DCS for conventional inorganics.

4.3.4.2 SCS. The SCS has the same components as the DCS. The SCS is analyzed with every analytical batch which does not include a pair of DCS. An analytical batch for conventional inorganics is defined as samples which are prepared or analyzed within a 24-hour period.

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4.3.4.3 Method Blank. For both aqueous and solid samples, an aliquot of deionized water taken through the entire analytical process serves as the method blank for conventional inorganic analyses. No solid matrix is incorporated in the method blank for solid samples.

A method blank must be analyzed with every analytical batch or with every 20 samples, whichever is more frequent. An analytical batch for conventional inorganics is defined as samples which are prepared or analyzed within a 24-hour period.

For conventional inorganic analyses, the method blank is analyzed in addition to the DCS and SCS (see Appendix A, Part 4).

4.3.4.4 Surrogates Spiked into Samples. Not applicable to Conventional Inorganic analyses.

4.4 Tracking of Laboratory QC Samples.

4.4.1 All samples analyzed are assigned a QC lot number. This lot number is used to store and track the QC information associated with a particular sample or group of samples. QC data are identified with the following components:

a) the QC category,
b) the QC type,
c) the QC lot number, and
d) the QC run number.

4.4.2 The QC Category and QC Type: Each test in LIMS is assigned to a QC category which specifies the QC type. The QC type is DCS, SCS, or Method Blank. Associated with each QC type is the component list for the QC category. Variations of the same test (e.g., VOA-HSL list, VOA-PP list, and VOA-Refinery list) may be assigned to the same QC category. QC categories are assigned to the major analytical groups as follows:

4.4.2.1 Volatile Organics (VOA). QC categories are assigned based on the detection system used. Separate QC categories are assigned for aqueous and solid matrices. Separate QC categories are assigned for

low-level and medium-level methods. An instrument specific identifier can be assigned to track VOA QC as instrument specific QC. QC categories and the components and spike levels for each QC type for volatile organics are specified in Appendix A1.

4.4.2.2 Extractable Organics. QC categories are assigned based on prep technique/detection system combination. Separate QC categories are assigned for aqueous and solid matrices. Separate QC categories are assigned for low-level and medium-level methods. QC categories and the components and spike levels for each QC type for extractable organics are specified in Appendix A2.

4.4.2.3 Metals. QC categories are assigned based on prep technique/detection system combination. Separate categories are assigned to aqueous and solid matrices. The categories for aqueous matrix are Dissolved Metals, Total Metals, and Total Recoverable Metals. The QC category Total Recoverable Metals is assigned for the solid matrix. QC categories and the components and spike levels for each QC type for metals are specified in Appendix A3.

4.4.2.4 Conventional Inorganics. QC categories are assigned based on the analytical method. Aqueous and solid matrices are typically tracked in the same QC category. Separate QC categories are assigned when there are significant differences in the sample prep. QC categories and the components and spike levels for each QC type for conventional inorganics are specified in Appendix A4.

4.4.3 The QC Lot: Each DCS pair which is analyzed within a QC category is assigned a unique QC lot number. This number follows the format YYMMMDD [letter] (e.g., 88MAR19A).

where: YY is the year,
MMM is the month, abbreviated as three letters,
DD is the day that the lot number is assigned, and
[letter] is a single letter (from A - Z) designating distinct QC lots for the day.

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This QC lot number is assigned to the DCS pair and all QC and environmental samples associated with these DCS. Typically, the same QC lot number is assigned to the DCS pair and the next 20 samples analyzed which have the same QC category.

The QC lot is assigned at the first step of the prep/analysis and tracks with the samples through analysis, data entry, and reporting. The QC lot is associated with the samples in LIMS during the data entry process.

4.4.4 The QC Run: Each analytical batch which is processed is assigned a QC run number which is used to track and store SCS and Blank results. The QC run is associated with a specific QC category and QC lot. The QC run number follows the format YYMMMDD [letter] (i.e., 88MAR15B)

where: YY is the year,
MMM is the month, as a three letter abbreviation,
DD is the day that the lot number is assigned, and
[letter] is a single letter (from A - Z) designating distinct QC runs for the day.

This QC run number is assigned to all samples analyzed within the analytical batch (QC samples plus environmental samples) and is also associated with a specific QC lot number.

The QC run is assigned at the first step of the prep/analysis and tracks with the samples through analysis, data entry, and reporting. The QC run is associated with the samples in LIMS during the data entry process.

Example:

Day 1 (March 4, 1988): 6 water samples analyzed for VOA-HSL. 20 samples have been analyzed since the last DCS were analyzed.

Day 2 (March 6, 1988): 7 water samples analyzed for VOA-PP.

Day 3 (March 7, 1988): 5 water samples analyzed f VOA-HSL.

Day 4 (March 9, 1988): 5 water samples analyzed for VOA-PP.

VOA-HSL and VOA-PP are both in the same QC category: 624-A. The lot numbers would be assigned as follows:

Day 1:
QC category	624-A
QC lot	88MAR04A
QC run	88MAR04A

These numbers are assigned to the DCS, the Method Blank, and the 6 samples analyzed on Day 1. A method blank must be analyzed with every batch. With this test, the method blank also contains surrogates and, therefore, also serves as an SCS, although an SCS is not required to be analyzed since DCS are included with the batch.

Day 2:
QC category	624-A
QC lot	88MAR04A
QC run	88MAR06A

These numbers are assigned to the 7 samples and the Method Blank/SCS analyzed on Day 2. DCS are not required because 20 samples have not been analyzed since the last set of DCS. A combination Method Blank/SCS is analyzed with the set to provide QC information for that day's analysis. A method blank must be analyzed with every batch. With this test, the method blank also contains surrogates and, therefore, also serves as the SCS.

Day 3:
QC category	624-A
QC lot	88MAR04A
QC run	88MAR07A

These numbers are assigned to the 5 samples and the Method Blank/SCS analyzed on Day 3. The rationale is exactly the same as outlined in Day 2.

Day 4:
QC category	624-A
QC lot	88MAR09A
QC run	88MAR09A

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These numbers are assigned to the DCS, the Method Blank/SCS and the 5 samples analyzed on Day 4. DCS are required because 20 samples have been analyzed since the last DCS pair were run.

4.5 Scheduling of Samples.

4.5.1 Samples should be scheduled for analysis such that all environmental samples and QC samples with the same QC run number are carried through the entire analytical process together.

4.5.2 In instances where it is impossible to analyze all samples with the same QC run number together, the QC samples should be analyzed with the first set of samples analyzed from that batch. QC samples, including method blanks, should be analyzed only once. (EPA-CLP and NJ Tier I work require that method blanks be analyzed on each of the instruments used to generate data for samples associated with the method blank. In these cases, method blanks may be analyzed multiple times. However, samples should be scheduled to minimize the number of method blank analyses required).

4.5.3 Samples should be scheduled for analyses according to the following priorities:

a) Meet holding times
b) Meet promised due date
c) Keep samples within a QC run together.

4.6 Analysis of Samples.

4.6.1 The QC program is structured so that decisions about the acceptability of the data can be made by the analyst at the time the data are generated.

4.6.2 Sample analysis should be conducted according to the following protocol:

4.6.2.1 Prepare the instrument for analysis by calibrating the instrument and verifying that the calibration is within acceptable guidelines (see SOP's related to specific method and equipment.)

4.6.2.2 Analyze all QC samples (method blanks, DCS and/or SCS) in the QC run. Determine if QC results are within acceptable limits. If data are outside control limits DO NOT PROCEED. CONTACT YOUR SUPERVISOR. Proceed with step (4.6.2.4). Analysts using autosamplers must verify that QC results are acceptable before leaving the instrument for unattended operation.

4.6.2.3 If QC samples are acceptable, proceed with analysis of other samples associated with the QC run.

4.6.2.4 If QC results are unacceptable, determine the source of the problem. Reevaluate standards, instrumentation, sample prep, etc. Each analyst should have a checklist to aid in identifying problems. Determine whether the analysis of samples should proceed using the following criteria:

a. Validate instrument operational setting, sensitivity, and linearity. If a problem is detected and corrected, reanalyze standard(s) and reanalyze QC samples. If QC data are within control limits, proceed with the analysis of the other samples in the QC run. If QC samples are still outside limits, continue to troubleshoot or go to Step b.

b. Validate that analytical standards are good. If a problem with the standards is detected, use a different standard, recalibrate, and reanalyze QC samples. If QC data are within control limits, proceed with analysis of the other samples in the QC run. If QC data are still outside of the limits, go to Step c.

c. Validate that sample prep was performed correctly. Check prep sheets for any anomalies. If no anomalies are found, reprep the samples. If samples cannot be reprepped due to lack of sample, analyze the other samples in the QC run and report data with a qualifier indicating that the Laboratory QC performed with the sample was out of

normal control limits. If sufficient sample remains, but holding times have expired. reprep samples unless otherwise required by the client.

If problems with the prep are discovered based on examination of prep sheets, notify the supervisor. The supervisor decides whether to reprep the samples based on the following criteria:

1. If the problems identified with the prep of the QC sample clearly affected only the QC sample and none of the other samples in the run (example: QC sample taken to dryness in the KD), a decision should be made not to reprep the samples, and the sample results can be reported without qualifiers.

2. If the problems identified could have potentially affected all of the samples in the QC run, the samples must be reprepped. If the samples cannot be reprepped for some reason, (i.e., inadequate sample volume) the sample results must be reported with a qualifier.

3. The entire episode, including reasons to support the final decision, must be documented on an anomaly form. The anomaly form is submitted to the QA Department. It is also forwarded, with the results, to the Program Administrator.

4.7 Calculation of QC Results.

4.7.1 DCS: The percent recovery of all spike components is calculated using the equation given in 4.2.1.1.

The precision of the DCS pairs is calculated by determining the RPD for each spike component using the equation given in 4.2.1.2.

4.7.2 SCS: The percent recovery of all spike components in the SCS is measured using the equation given in 4.2.2.

4.7.3 Method Blanks: The concentrations of target analytes are measured using quantitative techniques described in the method SOP.

4.7.4 Surrogate Spikes of Samples: The percent recovery of all surrogate spikes is calculated using the equation given in 4.2.2.

4.8 Interpretation of QC Results

4.8.1 Data acceptability is based upon the results of Laboratory Control Samples (DCS, SCS, and Method Blanks) unless otherwise negotiated with the client. Data acceptability based on criteria other than DCS, SCS, and Method Blanks falls under Matrix Specific QC (see SOP No. M-EQA-003).

4.8.2 The QC data from the DCS and SCS associated with a particular QC lot are compared to established control limits. QC data must fall within established control limits in order for the laboratory to be considered "in control" when samples from that QC lot were analyzed.

4.8.2.1 For single analyte tests, all DCS recovery and precision data and all SCS recovery data must be within established control limits in order for the laboratory to be considered "in control". Method blank values must be acceptable.

4.8.2.2 For organic multi-analyte tests, at least 80% of the average of each pair of DCS recovery data, and at least 80% of the DCS precision data, and at least 80% of the SCS recovery data must be within established control limits in order for the laboratory to be considered "in control". Method blank values must be acceptable. The 80% rule is tabulated below.

a.

# components in DCS	maximum numbers of values permitted outside control limits	
	average recovery	precision
1 - 4	0	0
5 - 9	1	1
10 - 14	2	2
15 - 19	3	3
20 - 24	4	4

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b.

# components in SCS	maximum number of values permitted outside control limits
1 - 4	0
5 - 9	1
10 - 14	2
15 - 19	3
20 - 24	4

4.8.2.3 For metals, all analytes requested by the client in the DCS associated with the samples must be within the control limits. Method blank values must be acceptable.

The DCS component lists for metals are divided into two categories:

a) Components which have been requested by the client to be analyzed in the samples associated with the DCS or SCS. The DCS or SCS counterpart of all analytes requested by the client must be within control limits in order for the laboratory to be considered "in control".

b) Components which are contained in the DCS or SCS but are not requested by the client. A maximum of three (3) analytes falling into this category may be outside control limits and the laboratory judged to be "in control". An anomaly form must be completed for these analytes to prevent analytical problems from going unresolved for a prolonged period of time.

Example: Two (2) projects are associated with the same DCS. Project A requires analyses of Ba, Ca, Cu, and V. Project B requires Cu, Ni, Na, Fe, and Zn.

In order for the lab to be "in control" and for analyses to proceed, the DCS values for Ba, Ca, Cu, V, Ni, Na, Fe, and Zn must be within control limits.

Of the remaining 13 metals in the DCS, at least 10 must also be within control limits.

4.8.3 Analytical data which are generated with DCS, SCS, and method blanks which fall within established control limits based on the criteria in 4.8.2, are judged to be in control. Data which are generated with DCS, SCS, or method blanks which fall outside of acceptance criteria are considered suspect and are reanalyzed or reported with qualifiers.

4.8.4 Sample results are accepted or rejected on the basis of the SCS, associated DCS and method blanks. If surrogates in the samples are outside of the control limits and the SCS is in control, the sample results are accepted and the surrogate recoveries are assumed to be affected by the matrix. In these cases, sample processing should be assessed to ensure that obvious problems with sample preparation and analysis (e.g. concentration of the sample to dryness, leaking septum) have been ruled out.

For certain projects, clients may specifically request sample reruns if the surrogates are outside specified limits in the samples (Lab limits or CLP limits). This is considered to be an element of Matrix Specific QC. Enseco policy duplicates the EPA-CLP program: a sample with surrogate recoveries outside the limits will be reanalyzed with the understanding that if, on reanalysis, surrogates fall within the limits, the lab reports the new data at no charge to the client. If, however, the second analysis duplicates the results from the first analysis, indicating a matrix effect, the client is charged for the second analysis. The same policy applies to matrix spike data (see SOP No. M-EQA-003).

4.9 Reporting of Laboratory Specific QC.

4.9.1 DCS, SCS, and method blank data are entered into LIMS with sample data. ALL QC data must be entered into LIMS, even if it fails the data acceptability criteria and samples are reanalyzed with new QC samples. The Blank QC Type contains method blank information for components typically found in a method blank. These components are lab-specific, but at a minimum, the components specified under Blank in Appendix A should be included in the Blank QC Type. Other compounds are

assumed to be ND (not detected). If the method blank is found to contain compounds other than those listed in the Blank QC type, they must be documented on the anomaly form and reported on the method blank data sheet.

4.9.2 Sample data must be reported with associated DCS, SCS, and method blank data. Sample data for organics and metals are not corrected for levels of analytes observed in the method blank. For conventional inorganic tests, the method SOP directs how the blank is treated. Method blank information is reported for all components in the analyte list for all organic and metals tests. Method blanks are not reported for most conventional inorganic tests, however, method blanks will be reported for Ion Chromatography, Cyanide, Phenol, TKN, Total Phosphorous, Fluoride by Distillation, Ammonia by Distillation, Chromium VI in soil, and Formaldehyde. QC data are reported in the QC section of the final report.

4.9.3 Report formats are shown in Appendix B.

5.0 RESPONSIBILITIES

5.1 The Analyst. The primary responsibility for implementation of the QC Program lies with the analyst. QC information needed for standard reports is generated, documented, and entered into LIMS by the analyst.

5.2 Management. The supervisors and managers who direct the analytical work are directly responsible for ensuring that all employees reporting to them are complying with the QC procedures set forth in this SOP.

5.3 QA Department. The QA Department is responsible for auditing the laboratory to ensure that the QC procedures set forth in this SOP are followed routinely. They are responsible for reviewing anomaly forms and initiating investigations of procedures and corrective actions, as appropriate.

5.4 Program Administrators/Technical Directors. Program Administrators and Technical Directors have the responsibility to convey to their client the quality of the data based on information contained on anomaly forms and in the LIMS QC database.

6.0 COMMENTS

As LIMS becomes more sophisticated, we will be tracking data based on both control limits (± 3 std. dev. units) and warning limits (± 2 std. dev. units). Enseco laboratories now have the ability to generate control charts using the QC data stored in LIMS. It is critical that ALL QC data, both good and bad, be entered into LIMS. The Method Blank database will be refined to allow entry of compounds other than common contaminants in an upcoming revision. The SOP will be updated at that time to reflect these changes.

7.0 DEFINITIONS

7.1 Quality Control (QC). The routine application of specific, well-documented procedures which ensures the generation of data of known and accepted quality which fulfill the objectives of the Quality Assurance Program.

7.2 Laboratory Control Sample (LCS). A well-characterized laboratory generated sample which is used to monitor the precision and accuracy of the analytical process or to assess the degree of laboratory contamination.

7.2 Duplicate Control Sample (DCS). A standard, control matrix which is spiked with a group of target compounds representative of the method analytes and is used to measure, on an ongoing basis, the precision and accuracy of a method, independent of matrix effects.

7.3. Single Control Sample (SCS). A standard, control matrix spiked with surrogate compounds appropriate to the method being used. In cases where no surrogate is available, the components of the DCS are used in the SCS. The SCS is used to monitor recovery with each analytical batch.

7.4 Method Blank. Reagents specific to a method which are carried through every aspect of the procedure to access the level of contamination which exists in the analytical system.

7.5 Analytical Batch. A group of samples considered to have been exposed to the same analytical conditions by virtue of the time frame in which they were analyzed.

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7.6 Ottawa Sand. This material is pure silica sand of particle size 20-30 mesh, specially prepared to conform to ASTM C190 and AASHTO T-132. (Ottawa is a trademark of Bellrose Silica). The laboratory must verify by analysis of the sand using the appropriate method that each lot of sand used is organic- and metal-free at the levels required for spiking target analytes as described in this SOP. Ottawa sand is available from Fisher Scientific.

APPENDIX A

Part 1

Laboratory Specific QC for Volatile Organics

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Method 601/8010
Halogenated Volatile Organics

QC Category	Aqueous	Solid
	601-A	8010-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
1,1-Dichloroethane	5.0	500
Chloroform	5.0	500
Bromodichloromethane	10.0	1,000
Trichloroethene	5.0	500
Chlorobenzene	5.0	500
SCS		
Bromochloromethane	30	3,000

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 5 g sample (wet weight)/10 mL methanol; 100 uL purged.

Method 602/8020
Aromatic Volatile Organics

QC Category	Aqueous	Solid
	602-A	8020-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Benzene	5.0	500
Toluene	5.0	500
1,3-Dichlorobenzene	5.0	500
Ethyl benzene	5.0	500
Total Xylenes**	5.0	500
SCS		
a,a,a,-Trifluorotoluene	30	3,000

Surrogates added to sample

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 5 g sample (wet weight)/10 mL methanol; 100 uL purged.

** Spiked as o-xylene; reported as Total Xylenes.

Method 603/8030
Acrolein/Acrylonitrile

QC Category	Aqueous	Solid
	603-A	8030-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Acrolein	25	2,500
Acrylonitrile	25	2,500

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 5 g sample (wet weight)/10 mL methanol; 100 uL purged.

Method 624/8240
Volatile Organics--GC/MS

QC Category	Aqueous	Low Level Solid	Medium Level Solid
	624-A	8240-SL	8240-S
		Spike Level	
DCS	Aqueous (ug/L)	Low Level Solid+ (ug/Kg)	Medium Level Solid* (ug/Kg)
1,1-Dichloroethene	50	50	5,000
Trichloroethene	50	50	5,000
Chlorobenzene	50	50	5,000
Toluene	50	50	5,000
Benzene	50	50	5,000
SCS			
1,2-Dichloroethane-d_4	50	50	5,000
4-Bromofluorobenzene (BFB)	50	50	5,000
Toluene-d_8	50	50	5,000

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

+ Assumes 5 g sample.
* Assumes 5 g sample (wet weight)/10 mL methanol; 100 uL purged.

Method 502.2

QC Category	5022-A
	Spike Level (ug/L)
DCS*	
Benzene	5
Vinyl Chloride	2
Carbon tetrachloride	5
1,2-Dichloroethane	5
Trichloroethene	5
1,1-Dichloroethene	7
1,1,1-Trichloroethane	10
1,4-Dichlorobenzene	10
Tetrachloroethene	5
Xylene (each isomer)	10
Chlorobenzene	10
Ethylbenzene	10
1,3-Dichloropropene	5
1,1,2,2-Tetrachloroethane	5
1,1,2-Trichloroethane	5
Total THMs	
Chloroform	10
Bromoform	10
Bromodichloromethane	10
Dibromochloromethane	10
trans-1,2-Dichloroethene	10
1,3-Dichlorobenzene	10
Methylene chloride	10
cis-1,2-Dichloroethene	10
1,2-Dichlorobenzene	10
Dibromomethane	10
1,1-Dichloropropane	10
Styrene	10
Chloromethane	10
Bromomethane	10
1,2,3-Trichloropropane	10
1,1,1,2-Tetrachloroethane	10
Chloroethane	10
2,2-Dichloropropane	10
o-Chlorotoluene	10
p-Chlorotoluene	10
Bromobenzene	10

* Any additional analytes which are added to the component list must be added to the DCS to comply with method requirements that ALL components analyzed for are contained in the QC check sample. The DCS serves as the QC check sample.

Method 502.2 Continued

QC Category — 5022-A

	Spike Level (ug/L)
SCS	
1-Chloro-4-fluorobenzene	5

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

Method 524.2

QC Category	5242-A
	Spike Level (ug/L)
DCS*	
Benzene	5
Vinyl Chloride	2
Carbon tetrachloride	5
1,2-Dichloroethane	5
Trichloroethene	5
1,1-Dichloroethene	7
1,1,1-Trichloroethane	10
1,4-Dichlorobenzene	10
Tetrachloroethene	5
Xylene (each isomer)	10
Chlorobenzene	10
Ethylbenzene	10
1,3-Dichloropropene	5
1,1,2,2-Tetrachloroethane	5
1,1,2-Trichloroethane	5
Total THMs	
Chloroform	10
Bromoform	10
Bromodichloromethane	10
Dibromochloromethane	10
trans-1,2-Dichloroethene	10
1,3-Dichlorobenzene	10
Methylene chloride	10
cis-1,2-Dichloroethene	10
1,2-Dichlorobenzene	10
Dibromomethane	10
1,1-Dichloropropane	10
Styrene	10
Chloromethane	10
Bromomethane	10
1,2,3-Trichloropropane	10
1,1,1,2-Tetrachloroethane	10
Chloroethane	10
2,2-Dichloropropane	10
o-Chlorotoluene	10
p-Chlorotoluene	10
Bromobenzene	10

* Any additional analytes which are added to the component list must be added to the DCS to comply with method requirements that ALL components analyzed for are contained in the QC check sample. The DCS serves as the QC check sample.

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Method 524.2

QC Category 5242-A

SCS	Spike Level (ug/L)
1,2-Dichloroethane-d_4	10
4-Bromofluorobenzene (BFB)	10
Toluene-d_8	10

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

Trihalomethanes

QC Category: THM-A

DCS

	Spike Level (ug/L)
Chloroform	2 ug/L
Bromoform	2 ug/L
Dibromochloromethane	2 ug/L
Bromodichloromethane	2 ug/L

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

APPENDIX A

Part 2

Laboratory Specific QC for Extractable Organics

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Method 604/8040
Phenols

QC Category	Aqueous	Solid
	604-A	8040-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
2-Nitrophenol	20	666
Phenol	20	666
2,4-Dinitrophenol	60	2,000
Pentachlorophenol	100	3,330
SCS		
2,4,6-Tribromophenol	100	3,330

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

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Method 606/8060
Phthalate Esters

QC Category	Aqueous	Solid
	606-A	8060-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Diethylphthalate	10	2500
Butylbenzylphthalate	1	250

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

Method 608/8080
Organochlorine Pesticides

QC Category	Aqueous	Low Level Solid	Low Level Solid with GPC	Medium Level Solid
	608-A	8080-S	8080-GPC-S	8080-MED-S

Spike Level

DCS	Aqueous (ug/L)	Low Level Solid (ug/Kg)	Low Level Solid with GPC (ug/Kg)	Medium Level Solid (ug/Kg)
Lindane	0.2	27	54	2000
Heptachlor	0.2	27	54	2000
Aldrin	0.2	27	54	2000
Dieldrin	0.5	67	134	5000
Endrin	0.5	67	134	5000
p,p'-DDT	0.5	67	134	5000
SCS				
Dibutylchlorendate	1	67	134	1000

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

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Method 608/8080
PCB only

QC Category	Aqueous	Solid
	PCB-A	PCB-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Aroclor 1254	5	167

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

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PCB in Oil

QC Category: PCBO-W

	Spike Level (ug/Kg)*
DCS	
Aroclor 1254	5,000
SCS	
Same as DCS	

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 1 g sample

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Method 8100
PNA's by GC/FID

QC Category	Aqueous	Solid
	610-GC-A	8100-S

DCS	Spike Level Aqueous (ug/L)	Solid* (ug/Kg)
Acenaphthene	50	2,500
Pyrene	50	2,500
Benzo(a)pyrene	50	2,500
SCS		
Orthoterphenyl	20	1,000

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 20 g sample (wet weight).

Method 612/8120
Chlorinated Hydrocarbons

QC Category	Aqueous	Solid
	612-A	8120-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
1,2,4-Trichlorobenzene	1.0	33.3

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989

Method 614/8140
Organophosphate Pesticides

QC Category	Aqueous	Solid
	614-A	8140-S
DCS	Spike Level Aqueous (ug/L)	Solid* (ug/Kg)
Phorate	10	330
Diazinon	10	330
Malathion	10	330
Parathion	10	330
Ethion	10	330
Methyl Parathion	10	330

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

Method 615/8150
Phenoxy Acid Herbicides

QC Category	Aqueous	Solid
	615-A	8150-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
2,4-D	5.0	170
2,4,5-TP (Silvex)	1.0	33
2,4,5-T	1.0	33
SCS		
2,4-Dichlorophenylacetic acid (DCAA)	5	100

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 50 g sample (wet weight)

Method 625/8270
Semivolatile Organics--GC/MS

QC Category	Aqueous	Low Level Solid	Low Level Solid with GPC	Medium Level Solid
	625-A	8270-S	8270-GPC-S	8270-MED-S
		Spike Level		
DCS	Aqueous (ug/L)	Low Level Solid* (ug/Kg)	Low Level Solid with GPC* (ug/Kg)	Medium Level Solid+ (ug/Kg)
Pentachlorophenol	200	6,670	13,300	200,000
Phenol	200	6,670	13,300	200,000
2-Chlorophenol	200	6,670	13,300	200,000
4-Chloro-3-cresol	200	6,670	13,300	200,000
4-Nitrophenol	200	6,670	13,300	200,000
1,2,4-Trichlorobenzene	100	3,330	6670	100,000
Acenaphthene	100	3,330	6670	100,000
2,4-Dinitrotoluene	100	3,330	6670	100,000
Pyrene	100	3,330	6670	100,000
N-Nitroso-di-N-propylamine	100	3,330	6670	100,000
1,4-Dichlorobenzene	100	3,330	6670	100,000
SCS				
Phenol-d_5	200	3,330	6670	200,000
2-Fluorophenol	200	3,330	6670	200,000
2,4,6-Tribromophenol	200	3,330	6670	200,000
Nitrobenzene-d_5	100	1,670	3330	200,000
2-fluorobiphenyl	100	1,670	3330	200,000
Terphenyl-d_{14}	100	1,670	3330	200,000

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight) + Assumes 1 g sample (wet weight)

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989

Method 8310
PNA's by HPLC

QC Category	Aqueous	Solid
	610-A	8310-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Naphthalene	5.0	1000
Fluorene	1.0	200
Pyrene	1.0	200
Benzo(a)pyrene	0.5	100
Indeno (1,2,3-cd) pyrene	0.5	100

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989

PCP & Tetrachlorophenol

QC Category	Aqueous	Solid
	PCP-A	PCP-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Pentachlorophenol	5	167
2,3,4,6-Tetrachlorophenol	5	167
SCS		
2,4,6-Tribromophenol	5	167

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

Marine Petroleum Hydrocarbons

QC Category	Aqueous	Solid
	MHYD-A	MHYD-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Fuel Oil #2	1,000	50,000
SCS		
Orthoterphenyl	20	1,000

Surrogates added to samples

Same as SCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 20 g sample (wet weight)

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989

Method 504
DBCP and EDB

QC Category: 504-A

DCS	Spike Level Aqueous (ug/L)
DBCP	0.25
EDB	0.25

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989

Method 619
Triazines--HPLC

QC Category	Aqueous	Solid
	619-LC-A	8190-LC-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Simazine	10	333
Cyanazine	10	333
Atrazine	10	333
Prometon	10	333
Propazine	10	333
Prometryn	10	333

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

Method 619
Triazines--GC

QC Category	Aqueous	Solid
	619-GC-A	8190-GC-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Simazine	10	333
Cyanazine	10	333
Atrazine	10	333
Prometon	10	333
Propazine	10	333
Prometryn	10	333

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

Method 632
Carbamates--HPLC

QC Category	Aqueous	Solid
	632-A	632-S
	Spike Level	
DCS	Aqueous (ug/L)	Solid* (ug/Kg)
Oxamyl	50	1,665
Baygon	100	3,330
Furadan	100	3,330
Carbaryl	50	1,665
Diuron	5	167

SCS

Same as DCS

Surrogates added to samples

None

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 30 g sample (wet weight)

Dioxins & Furans

QC Category	Aqueous	Solid
	DXNFUR-A	DXNFUR-S
	Spike Level	
DCS	Aqueous (ng)/sample	Solid (ng)/sample
2,3,7,8-TCDF	10	10
2,3,4,7,8-PECDF	10	10
1,2,3,4,7,8-HXCDF	10	10
1,2,3,4,6,7,8-HPCDF	10	10
1,2,3,4,5,6,7,8-OCDF	50	50
2,3,7,8-TCDD	10	10
1,2,3,7,8-PECDD	10	10
1,2,3,4,7,8-HXCDD	10	10
1,2,3,4,6,7,8-HPCDD	10	10
1,2,3,4,5,6,7,8-OCDD	50	50
SCS		
Same as DCS		
Surrogates added to samples		
37 Cl-2,3,7,8-TCDD	10	10

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

TCDF & TCDD

QC Category	Aqueous	Solid
	TCDD-A	TCDD-S
	Spike Level	
DCS	Aqueous (ng)/sample	Solid (ng)/sample
2,3,7,8-TCDF	10	10
2,3,7,8-TCDD	10	10

SCS

Same as DCS

Surrogates added to samples

37 Cl-2,3,7,8-TCDD	10	10

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

SOP No.:	Revision No.:	Effective Date:
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High Resolution Dioxins & Furans

QC Category	Aqueous	Solid
	DXNFUR-HR-A	DXNFUR-HR-S
	Spike Level	
DCS	Aqueous (ng)/sample	Solid (ng)/sample
2,3,7,8-TCDF	0.20	0.20
1,2,3,7,8-PECDF	0.50	0.50
1,2,3,4,7,8-HXCDF	0.50	0.50
1,2,3,4,6,7,8-HPCDF	0.50	0.50
1,2,3,4,5,6,7,8-OCDF	1.0	1.0
2,3,7,8-TCDD	0.20	0.20
1,2,3,7,8-PECDD	0.50	0.50
1,2,3,4,7,8-HXCDD	0.50	0.50
1,2,3,4,6,7,8-HPCDD	0.50	0.50
1,2,3,4,5,6,7,8-OCDD	1.0	1.0
SCS		
Same as DCS		
Surrogates added to samples		
37 Cl-2,3,7,8-TCDD	1.0	1.0

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

High Resolution TCDF & TCDD

QC Category	Aqueous	Solid
	TCDD-HR-A	TCDD-HR-S
	Spike Level	
DCS	Aqueous (ng)/sample	Solid (ng)/sample
2,3,7,8-TCDF	0.20	0.20
2,3,7,8-TCDD	0.20	0.20

SCS

Same as DCS

Surrogates added to samples

37 Cl-2,3,7,8-TCDD	1.0	1.0

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

APPENDIX A

Part 3

Laboratory Specific QC for Metals

ICP Metals

QC Category	Aqueous	Solid
	ICP-AD ICP-AT ICP-AR	ICP-S

DCS	Spike Level Aqueous (mg/L)	Solid* (mg/Kg)
Aluminum	2.0	200
Antimony	0.5	50
Arsenic	0.5	50
Barium	2.0	200
Beryllium	0.05	5.0
Cadmium	0.05	5.0
Calcium	100	10,000
Chromium	0.2	20
Cobalt	0.5	50
Copper	0.25	25
Iron	1.0	100
Lead	0.5	50
Magnesium	50	5,000
Manganese	0.5	50
Nickel	0.5	50
Potassium	50	5,000
Silver	0.05	5.0
Sodium	100	10,000
Vanadium	0.5	50
Zinc	0.5	50

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 1 g sample (wet weight)

Graphite Furnace Metals--Mix 1

	QC Category		Spike Level	
DCS	Aqueous	Solid	Aqueous (mg/L)	Solid* (mg/Kg)
Arsenic	AS-FAA-AD AS-FAA-AT AS-FAA-AR	AS-FAA-S	0.04	4
Lead	PB-FAA-AD PB-FAA-AT PB-FAA-AR	PB-FAA-S	0.02	2
Selenium	SE-FAA-AD SE-FAA-AT SE-FAA-AR	SE-FAA-S	0.01	1
Thallium	TL-FAA-AD TL-FAA-AT TL-FAA-AR	TL-FAA-S	0.05	5

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 1 g sample (wet weight)

Graphite Furnace Metals--Mix 2

	QC Category		Spike Level	
DCS	Aqueous	Solid	Aqueous (mg/L)	Solid* (mg/Kg)
Cadmium	CD-FAA-AD CD-FAA-AT CD-FAA-AR	CD-FAA-S	0.002	0.2
Silver	AG-FAA-AD AG-FAA-AT AG-FAA-AR	AG-FAA-S	0.002	0.2

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 1 g sample (wet weight)

SOP No.:	Revision No.:	Effective Date:
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Mercury by Cold Vapor--AA

QC Category	Aqueous	Solid
	HG-CVAA-AT	HG-CVAA-S
	Spike Level	
DCS	Aqueous (mg/L)	Solid* (mg/Kg)
Mercury	0.001	0.5

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 0.2 g sample (wet weight)

APPENDIX A

Part 4

Laboratory QC for Conventional Inorganics

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989

Conventional Inorganics

Test/DCS Component	QC Category		Spike Level*
	Aqueous	Solid	(ppm unless noted)
Ammonia as N	NH3-A		2.0
Ammonia as N, Distilled	NH3-DIST-A		2.0
Hardness as $CaCO_3$, Tit.	HARD-A		300
Iodide	I-A		0.50
Fluoride	F-A		10
Fluoride, Distilled	F-DIST-A		10
MBAS	MBAS-A		0.50
Cyanide, Total	CN-A		0.10
Cyanide, Free	CNW-A		0.10
Dissolved Cyanide	CN-A		0.10
Dissolved Sulfide	S-A		0.50
Chemical Oxygen Demand	COD-A	COD-S	125
Chloride	CL-A		20
Biochemical Oxygen Demand	BOD-A		200
Bromide	BR-A		0.50
Hexavalent Chromium	CR6-A		0.05
Hexavalent Chromium		CR6-S	5
Nitrate as N, Colorimetric	NO3-A		2.0
Nitrite as N, Colorimetric	NO2-A		0.10
Orthophosphate as P	PO4-A		0.25
Phenolics	PHEN-A		0.20
Purgeable Organic Carbon	POC-A		10
Purgeable Organic Halogen	POX-A		100 ppb
Reactive Cyanide		CNR-S	100
Reactive Sulfide		SR-S	100
Residual Chlorine	CL2-A		2.0
Sulfate	SO4-A		100
Thiocyanate	SCN-A		3.0
Thiosulfate	S2O3-A		3.0
Total Alkalinity as $CaCO_3$	ALK-A		190
Total Dissolved Solids	TDS-A		1,400
Total Kjeldahl Nitrogen as N	TKN-A		3.0
Total Organic Carbon	TOC-A	TOC-S	25
Total Organic Halogen	TOX-A	TOX-S	100 ppb
Total Phosphorus as P	TPHOS-A		0.25
Total Solids	TS-A		1,400
Total Sulfide	S-A		0.5
Total Sulfide		S-S	100
Total Suspended Solids	TSS-A		45
pH	PH-A		9.0 units
Specific Conductance @ 25 C	COND-A		1,860 umhos/cm
Turbidity	TURB-A		1.5 NTU

* Aqueous and solid samples spiked identically unless otherwise specified in a method SOP. Spike levels are nominal and may vary if spike solutions are obtained from outside vendors. Spike levels should be within the same order of magnitude as those specified.

Ion Chromatography

DCS	QC Category	Spike Level Aqueous (mg/L)
Fluoride	F-IC-A	5
Chloride	CL-IC-A	100
Nitrite as N	NO2-IC-A	2
Bromide	BR-IC-A	20
Nitrate as N	NO3-IC-A	20
Sulfate	SO4-IC-A	200
Orthophosphate as P	PO4-IC-A	20

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989

Method 413.1,2
Oil and Grease

DCS	Method	QC Category		Spike Level	
		Aqueous	Solid	Aqueous (mg/L)	Solid* (mg/Kg)
Aromatic Ref. Oil (1)	Aromatic	O&G-AR-A	O&G-AR-S	50	1000
Reference Oil (2)	Gravimetric	O&G-G-A	O&G-G-S	50	1000
Reference Oil (3)	IR	O&G-IR-A	O&G-IR-S	5	50+

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

(1) As specifed in LP-RMA-1044 and LP-RMA-1045
(2) As specified in Method 413.1
(3) As specified in Method 413.2

* Assumes 50 g sample (wet weight).

+ Spike level may be adjusted to account for variation in instrument sensitivity. Alternate spike level must be within 5 times the reporting limit.

C

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989

Method 418.1
Total Petroleum Hydrocarbons by IR

QC Category	Aqueous	Solid
	TPH-IR-A	TPH-IR-S
	Spike Level	
DCS	Aqueous (mg/L)	Solid* (mg/Kg)
Reference Oil (per Method 418.1)	5.0	50+

SCS

Same as DCS

Blank components in LIMS

At a minimum, the Blank QC Type should contain the test components frequently identified in the method blank.

* Assumes 50 g sample (wet weight)

+ Spike level may be adjusted to account for variation in instrument sensitivity. Alternate spike level must be within 5 times the reporting limit.

APPENDIX B

Report Formats

SOP No.:	Revision No.:	Effective Date:
M-EQA-002	2.0	May 15, 1989

QC LOT ASSIGNMENT REPORT
Volatile Organics by GC

Laboratory Sample Number	QC Matrix	QC Category	QC Lot Number (DCS)	QC Run Number (SCS/BLANK)
003338-0001-SA	AQUEOUS	601-A	12 APR 89-A	12 APR 89-A
003338-0001-SA	AQUEOUS	602-A	12 APR 89-A	12 APR 89-A
003338-0001-MS	AQUEOUS	601-A	12 APR 89-A	12 APR 89-A
003338-0001-MS	AQUEOUS	602-A	12 APR 89-A	12 APR 89-A

DUPLICATE CONTROL SAMPLE REPORT
Volatile Organics by GC

Analyte	Concentration Spiked	Measured DCS1	Measured DCS2	Measured AVG	Accuracy Average(%) DCS	Accuracy Average(%) Limits	Precision (RPD) DCS	Precision (RPD) Limits
Category: 601-A								
Matrix: AQUEOUS								
QC Lot: 12 APR 89-A								
Concentration Units: ug/L								
1,1-Dichloroethane	5.0	5.76	5.95	5.86	117	60-140	3.4	20
Chloroform	5.0	5.20	5.28	5.24	105	60-140	1.9	20
Bromodichloromethane	10	9.75	9.83	9.79	98	60-140	0.0	20
Trichloroethene(-ylene)	5.0	5.13	5.03	5.08	102	60-140	2.0	20
Chlorobenzene	5.0	5.03	5.02	5.02	100	60-140	1.0	20
Category: 602-A								
Matrix: AQUEOUS								
QC Lot: 12 APR 89-A								
Concentration Units: ug/L								
Benzene	5.0	5.30	5.50	5.40	108	77-123	3.7	20
Toluene	5.0	5.50	ND	NC	NC	77-123	NC	20
Chlorobenzene	5.0	5.50	6.00	5.75	115	77-123	8.7	20
Ethyl benzene	5.0	4.00	4.56	4.28	85	77-123	13	20
o-Xylene	5.0	3.00	3.00	3.00	60	77-123	0.0	20
1,3-Dichlorobenzene	5.0	5.00	5.00	5.00	100	77-123	0.0	20

ND = Not detected.
NC = Not calculated; calculation not appropriate.
NA = Not applicable.

Calculations are performed before rounding to avoid round-off errors in calculated results.

SINGLE CONTROL SAMPLE REPORT
Volatile Organics by GC

Analyte	Concentration Spiked	Concentration Measured	Accuracy(%) SCS	Accuracy(%) Limits
Category: 601-A Matrix: AQUEOUS QC Lot: 12 APR 89-A QC Run: 12 APR 89-A Concentration Units: ug/L				
Bromochloromethane	30.0	31.4	105	20-160
Category: 602-A Matrix: AQUEOUS QC Lot: 12 APR 89-A QC Run: 12 APR 89-A Concentration Units: ug/L				
a,a,a-Trifluorotoluene	30.0	30.0	100	20-160

Calculations are performed before rounding to avoid round-off errors in calculated results.

METHOD BLANK REPORT
Volatile Organics by GC

Test: 601-A
Matrix: AQUEOUS
QC Lot: 12 APR 89-A QC Run: 12 APR 89-A

Analyte	Result	Units	Reporting Limit
Chloromethane (Methyl chloride)	ND	ug/L	5.0
Bromomethane	ND	ug/L	5.0
Vinyl chloride	ND	ug/L	1.0
Chloroethane	ND	ug/L	5.0
Methylene chloride	ND	ug/L	5.0
1,1-Dichloroethene	ND	ug/L	0.50
1,1-Dichloroethane	ND	ug/L	0.50
1,2-Dichloroethene (total)	ND	ug/L	0.50
1,1,2-Trichloro-2,2, 1-trifluoroethane	ND	ug/L	1.0
1,2-Dichloroethane	ND	ug/L	1.0
1,1,1-Trichloroethane	ND	ug/L	0.50
Carbon tetrachloride	ND	ug/L	0.50
Bromodichloromethane	ND	ug/L	1.0
1,2-Dichloropropane	ND	ug/L	1.0
trans-1,3-Dichloropropene	ND	ug/L	1.0
Trichloroethene(-ylene)	ND	ug/L	0.50
Dibromochloromethane	ND	ug/L	1.0
cis-1,3-Dichloropropene	ND	ug/L	2.0
1,1,2-Trichloroethane	ND	ug/L	1.0
EDB (1,2-Dibromoethane)	ND	ug/L	2.0
Bromoform	ND	ug/L	5.0
1,1,2,2-Tetrachloroethane	ND	ug/L	1.0
Tetrachloroethene	ND	ug/L	0.50

METHOD BLANK REPORT
Volatile Organics by GC

Test: 602-AP
Matrix: AQUEOUS
QC Lot: 12 APR 89-A QC Run: 12 APR 89-A

Analyte	Result	Units	Reporting Limit
Benzene	ND	ug/L	0.50
Toluene	ND	ug/L	0.50
Chlorobenzene	ND	ug/L	0.50
Ethyl benzene	ND	ug/L	0.50
Xylenes (total)	ND	ug/L	1.0
1,3-Dichlorobenzene	ND	ug/L	0.50
1,4-Dichlorobenzene	ND	ug/L	0.50
1,2-Dichlorobenzene	ND	ug/L	0.50

STANDARD
OPERATING
PROCEDURE

Subject or Title: **Corrective Action Report**

SOP No.: LP-RMA-0022 | Revision No.: 1 | Effective Date: 8/9/88

Supersedes:

1.0 Purpose

1.1 To document and communicate quality control problems encountered during analysis.

1.2 To initiate an investigation of quality control problems.

1.3 To implement corrective actions designed to ensure that quality control problems will be minimized in future analyses.

1.4 To allow follow-up of quality control problems at a later date.

2.0 Policies

2.1 All errors, deficiencies, and out-of-control situations encountered with analytical standards, client samples and laboratory-generated quality control samples must be documented on the warning/out-of-control form contained in this S.O.P.

2.2 An investigation must be initiated immediately upon discovering a problem may exist.

2.3 Upon completing the investigation, corrective actions are undertaken by, and communicated to, the appropriate staff members in the department(s) in which the problem occurred.

Prepared by: Gary Tolf	Date: 8/9/88
Management Approval: Kathy Carlberg	Date: 8/15/88
QA Officer Approval: Gary Tolf	Date: 8/15/88

2.4 The warning/out-of-control form must be signed and dated by the staff member(s) investigating the problem, a supervisor within the department that encountered the problem, and a member of the quality assurance department.

2.5 A copy of the signed form must be placed with the raw data and project folders of the affected samples, and provided to the Quality Assurance Department for archiving (it is highly recommended that the manager of the affected department also maintain a copy for communication and follow-up within the department), as described in 5.2 and 5.3.

3.0 Safety Issues

None

4.0 Procedure

4.1 Identification of Out-of-Control Situation

4.1.1 Situations which alert laboratory personnel that an out-of-control situation exists and corrective action may be necessary include, but are not limited to:

- QC data are outside the warning or acceptable limits for precision and accuracy,
- Blanks, LCS or SCS control samples contain contaminants above acceptable levels,
- There are unusual changes in detection limits,
- Deficiencies are detected by the Quality Assurance department during internal or external audits or from the results of performance evaluation samples,
- Inquiries concerning data quality are received from clients or other Enseco facilities.

4.2 Documentation of the Out-of-Control Situation

4.2.1 Briefly, but informatively describe the problem encountered on the warning/out-of-control form contained in this S.O.P. For example:

"The percent recovery for the Cyanide LCS1, QC lot 880915A, was 60%. The accuracy control limits are 75% - 125%".

Rather than,

"The LCS was out".

"The precision for Phenol in the BNA LCS, QC lot 880915A, was 66.7. The precision control limit is 42.0".

Rather than,

"The Phenol precision was no good".

"Aldrin was found in the OCP SCS, QC lot 880915A".

Rather than,

"The SCS was contaminated".

4.2.2 List all samples affected by the problem. Specify whether the problem affected only the laboratory quality control samples, the client samples, or both.

4.3 Document the investigation of the problem.

4.3.1 List each step of the analytical procedure which was investigated, and the result/conclusion. It is acceptable to document that no definitive conclusion can be drawn, however, specific follow-up investigations will then be required as a corrective action to ensure the problem does not persist.

4.3.2 List any additional samples and/or QC lots found during the investigation to be affected by the problem.

4.4 Document the corrective actions taken to resolve the problem.

4.4.1 List any immediate steps taken to resolve the problem with the affected samples, such as repreparation or reanalysis.

4.4.2 List the actions taken to ensure that the problem does not affect future analyses. Examples include (but are not limited to): Maintenance of laboratory equipment, specific changes in standard operating procedures, additional training of personnel, and the purchasing of reagents or standards from new sources/vendors.

4.5 Communicate the resolution of the problem and any necessary follow-up to appropriate staff members.

4.5.1 The warning/out-of-control form is signed and dated by the staff member investigating the problem.

4.5.2 The warning/out-of-control form is signed and dated by a supervisor within the department that encountered the problem.

4.5.3 The warning/out-of-control form is signed and dated by a member of the Quality Assurance Department.

4.5.4 The warning/out-of-control form is signed by a client manager (optional).

4.5.5 A copy of the warning/out-of-control form is:

- Placed with the raw data and project folders of any affected samples,
- Provided to the Quality Assurance Department for archiving and,
- Provided to the client manager when requested.

4.5.6 The problem and it's resolution is communicated to additional staff members in the affected department(s) at the next scheduled group meeting.

5.0 Responsibilities

5.1 The staff member (technician, analyst/chemist, supervisor, quality assurance personnel, client manager, technical director, et al) who encounters an error, deficiency or out-of-control situation is responsible for immediately documenting the problem as described in 4.2 above.

5.2 The supervisor/manager in the department where the problem occurred is responsible for:

- The timely investigation of the problem,
- The documentation of corrective actions,
- Notification to the Quality Assurance Department and affected client manager(s),
- Ensuring corrective actions are implemented and appropriate follow-up in undertaken,
- Ensuring that a copy of the warning/out-of-control form is placed with the raw data and provided to the appropriate personnel as described in 4.5.5 and 4.5.6 above and,
- Communicating the problem and its resolution to additional staff members in their department.

5.3 The Quality Assurance Department is responsible for:

- The timely acknowledgement of any warning/out-of-control forms after completion, archival of the signed forms in the project folder and QA files, and
- The timely approval of any S.O.P. changes necessitated by corrective actions.

5.4 The client manager is responsible for communicating to the client the affect a problem may have on the analysis of their samples (verbally and/or in the final report).

6.0 Comments

6.1 Laboratory supervisors and managers should encourage the use of the warning/out-of-control form by:

- Reiterating that Quality Control is the responsibility of all staff members and,

- The prompt addressing of major and minor problems brought to their attention.

Corrective Action Report

SOP No.: LP-RMA-0022 Revision No.: 1 Effective Date: 8/9/88

WARNING/OUT-OF-CONTROL FORM

QC Lot _____

Associated Samples _____

PROBLEM: (Briefly decribe problem)

Analyst:
Date:

RESULTS/CONCLUSIONS of the Investigation:

Analyst:
Supervisor:
Date:

CORRECTIVE ACTIONS (including follow-up)

Supervisor:
QA Approval:
Date:

WARNING/OUT-OF-CONTROL FORM

QC Lot _____

Associated Samples _____

PROBLEM: (Briefly decribe problem)

Analyst:
Date:

RESULTS/CONCLUSIONS of the Investigation:

Analyst:
Supervisor:
Date:

CORRECTIVE ACTIONS (including follow-up)

Supervisor:
QA Approval:
Date:

STANDARD
OPERATING
PROCEDURE

Subject or Title:
CLP SAMPLE RECEIPT AND LOGGING

SOP No.:	Revision No.:	Effective Date:
LP-RMA-5001	Original	10/1/90

Supersedes: None

ENSECO PROPRIETARY INFORMATION STATEMENT

1. Purpose:

   To assure traceability of the samples while in possession of the laboratory and to create analyses records in the laboratory computer for notification to lab analysts.

2. Policies:

   To receive and handle samples under the Chain-of-Custody procedures described in the Statement of Work for Organic Analysis, Exhibit F.

3. Safety: Not Applicable

Prepared by: Jan Redenbarger	Date: 11/7/90
Management Approval: Gary Lof	Date: 11-12-90
QA Officer Approval: Jan Redenbarger	Date: 11/7/90

4. Procedures

4.1 Check cooler for radiation

4.2 Check condition of shipping container upon receipt

4.3 Check custody seals: intact/not intact

4.4 Check for presence or absence of EPA Chain-of-Custody Traffic Report or SAS packing list.

4.5 Verify case number with RAS or SAS booking. (Bookings are scheduled with RMAL by Sample Management Office (SMO)). If case number is not scheduled with RMAL call SMO (environmental program coordinator by region) for clarification.

4.6 Use CLP or EPA sample receipt checklist (See figures 1,2, and 3).

4.7 Unpack cooler

4.7.1 Check condition of samples.

4.7.2 Verify Chain of Custody, Traffic Report or SAS packing list, tags with sample labels.

4.8 Air bill or air bill sticker numbers should be on Chain-of-Custody Traffic Report/SAS packing list, tags should also be on Chain-of-Custody.

4.9 Sign, date, and time Chain-of-Custody, Traffic Report/SAS packing list, and air bill if no discrepancies are found.

4.9.1 If discrepancies are found contact SMO. Contact the Environmental Program Coordinator by region. Document agreement among the forms and any discrepancies found on sample receipt checklist with date, time, and initials.

4.10 Check paper work (Chain-of-Custody, Traffic Report or packing list for designated QC (matrix spike and duplicate). If QC is not indicated sample custodian will select QC.

4.11 Select SDG number

The SDG number is the lowest sample number in the first group of samples received under the SDG. In determining lowest sample number, consider both alphabetic and numeric characters in the sample number.

A Sample with a lower number could arrive in a later sample shipment so be aware that the SDG number may not necessarily be the lowest sample number. The SDG number appears on all data deliverables.

4.12 Assign QC number (EPA Organic), pull RMA's project number through LIMS by client (SMO*) return (see figure 4).

- Region
- Program
- Project
- Add Project

- or duplicate from project with existing region (by using the Log-in Book for EPA Organic).

Assign QC number (CLP Organic), pull RMA's project number through LIMS (by existing client in the right program). PA will set up all new clients in LIMS.

4.13 Stamp information on project folder (see figure 5)

- QC number
- Contact number
- Controller
- Turnaround
- Region
- Price
- Parameters
- Others
- No type
- Date received
- Date due

(Note all rush projects on folder)

4.14 Label samples with project number, plus EPA or CLP initials so that samples are located in the correct area of the cooler.
Example: 011829 EPA or 011829 CLP

4.15 Log-in to LIMS by matrix (aqueous or soil)

Each matrix is a separate project for EPA or CLP.

4.16 Print out group code (EPA)
- sample list

Print out group code (CLP)
- sample list
- acceptance letter

4.17 Take picture of samples for project folder. (Take separate picture of broken samples).

4.18 Complete project folder (see figure 6).
- SDG package which contains:
  - SDG cover sheet
  - original Traffic Report/SAS packing list
  - mail to SMO

4.18.1 If project has LIMS print out
- SDG cover sheet
- copy Traffic Report/SAS packing list
- Chain-of-Custody
- air bill
- check list
- picture
- (CLP) cross-reference of samples
- (CLP) acceptance letter
- write project number, case number, number of sample matrix and parameters on (EPA) Chain-of-Custody. Special instruction in LIMS, if any.
- Special instructions in LIMS, if any.

4.19 Record projects in EPA Organic or CLP Organic Log Book

4.20 Give project folder to PA

PA will review project folders and put away in designated area.

4.21 Put sample away in walk-in cooler or refrigerator; volatile samples are stored in separate refrigerators by matrix.

4.22 Write sample project number and shelf number on board.

5. Responsibilities:

Sample Custodian-Responsible for receiving the EPA samples (logging, handling, and storage).

6. Definitions:

6.1 CLP - Contract Laboratory Program

6.2 EPA - Environmental Protection Agency

6.3 LIMS - Laboratory Information Management System, commercial and custom software programs designed to perform calculations, check results, generate reports, and ensure data integrity and security.

6.4 PA - Program Administrator

6.5 SAS - Special Analytical Services

6.6 Sample - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

6.7 Sample Delivery Group (SDG) - a unit within a single Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a Case, received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, which ever occurs first:

- Case; or
- Each 20 field samples within a Case; or
- Each 14-day calendar period during which field samples in a Case or SDG.

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory.

6.8 Sample Number (EPA Sample Number) - a unique identification number designated by EPA for each sample. The EPA sample number appears on the sample Traffic Report which documents information on that sample.

6.9 Special instructions - typed instructions in LIMS to the operations groups and analysts that are necessary to complete the work and can not by indicated by using one of the computer tests.

6.10 Traffic Report (TR) - an EPA sample identification form filled out by the sampler, which accompanies the sample during shipment to the laboratory and which documents sample condition and receipt by the laboratory.

Figure 1

EPA SAMPLE RECEIPT CHECKLIST

YES	NO	
____	____	1a. Chain of Custody present?
____	____	1b. Chain of Custody filled out correctly?
____	____	2a. Traffic Reports or Packing List present?
____	____	2b. Traffic Reports or Packing List filled out correctly?
____	____	3a. Tags present on sample containers?
____	____	3b. Tags filled out correctly?
____	____	4. Custody seals present on sample cooler?
____	____	5. Custody seals present on sample containers?
____	____	6. Sample labels present on sample containers?
____	____	7. Samples intact?
____	____	8. Samples cold upon receipt?
____	____	9. Chain of Custody, Traffic Reports (or Packing List), Tag, and Label agree?
____	____	10. Number and type of samples and parameters agree with booking?

If NO is checked, note action taken:

If SMO is contacted, note date and person contacted:

Checklist completed by:

Name: _____

Date: _____

CLP SAMPLE RECEIPT CHECKLIST

YES	NO		
_____	_____	1a.	Chain of Custody present?
_____	_____	1b.	Chain of Custody filled out correctly?
_____	_____	2.	Custody seals present on sample cooler?
_____	_____	3.	Custody seals present on sample containers?
_____	_____	4.	Sample labels present on sample containers?
_____	_____	5.	Samples intact?
_____	_____	6.	Samples cold upon receipt?
_____	_____	7.	Chain of Custody and Label agree?

If NO is checked, note action taken:

Checklist completed by:

Name: _____

Date: _____

Figure 3

RMAL Industrial Sample Checklist Revision 2.0 March, 1989

Project #: _____ Date/Time Received: _____

mpany Name & Sampling Site : _____

*Cooler # (s): _____ _____ _____ _____ * Place copy of airbill inside all non-RMAL coolers. Describe here.

Temperatures: _____ _____ _____ _____

UNPACKING & LABELING CHECK POINTS:	Y	N	INITIALS
1. Radiation Checked; (record reading if > 15 mr):	___	___	_____
2. Cooler seals Intact:	___	___	_____
3. Chain of Custody Present:	___	___	_____
4. Bottles broken or leaking (comment if Y): **- photograph broken bottles-**	___	___	_____
5. Containers labeled (comment if N):	___	___	_____
6. Aqueous: ___ Soil: ___ Waste: ___			_____
7. Chain of Custody signed with date, time & lab:	___	___	_____
8. CoC agrees with bottle count (comment if N):	___	___	_____
9. CoC agrees with labels (comment if N):	___	___	_____
10. VOA samples filled completely (comment if N):	___	___	_____
11. VOA samples preserved:	___	___	_____
12. Sediment present in "D" bottles:	___	___	_____
13. Short holding times:	___	___	_____
14. Matrix QC verified:	___	___	_____
15. Multi phase samples present (comment if Y): **- photograph multiphase samples -**	___	___	_____
16. Clear picture taken & labeled:	___	___	_____
17. Clear picture of sub samples taken:	___	___	_____

18. Comments; include action taken to resolve discrepancies / problems. Include a hardcopy of VAX mail or extra paper if more space is needed.

_____

_____

_____

_____ Sign & Date: _____

Figure 4

```
Log-in

Limsmenu
Project/Data Maintenance
Screen Client
    EPA Organic (SMO*)
    CLP Organic
    Client (SMO*)    Return
    Soft Key (F-19)    Program Return

    Shift ~ Return
    (Program Search)
    Soft Key    Project    (F19)    Return
    Soft Key    Add Project    (F11)    Return

    Name Case #    SDG #    Final SDG #
    Return
    Type None
    Return
    Case #    SDG #    SAS #   Level
    Return

Description:  (Example) two waters, full HSL.
    Return
Soft key (Page 2) F14
Calculate holding times from collection dates?   N
Return
Actual start date
    Return
Receipt    date    time
    Return
         Do
More functions (F17)
Soft key    accept delivery    (F11)
    Do
    F-10
    Group Code    return
    Add Group Code (F-11)
    Description Example: samples 1-2 (MS,SD)
         Do

    Add test    (F12)
         Example (625-RAS-A)    Do
         (F12)  (624-RAS-A)    Do
         (F12)  (608-RAS-A)    Do
    (F10)
    Sample Return
    Add Sample (F11)
    Aliquot (SA) Sample    Return    Group Code A    Return
    Client Description Example (MHT58)    Return
    Collection Date:  "Return Time"    Return
    Receipt Date:  "Return Time"    Return
    Lab Description:  Example (3-11, 2-12, and 3-12)
```

Figure 4 (con't)

```
    Return
    Do
    For QC
    Duplicate Sample    (F14)
Ctrl U (0001) Aliquot (MS) Return    Group Code (A)    Return
    Do
    Duplicate Sample    (F14)
Ctrl U (to sample number indicated for QC)
    Return
Aliquot (MS) Matrix Spike
Group Code (if necessary)
Client Description    Return
    Do
    Duplicate Sample    (F14)
Ctrl U
    Return
Aliquot (SD) matrix spike duplicate
        Do
    Duplicate Sample    (F14)
Change
Aliquot (SA)    Return
Client Description
Collection Date    Return    Time    Return
Lab Description

        Do
    F10
    Group Code    Return
    Ctrl R    No-Yes    Return
    (copy of Group Code)
    F10 Samples
    Ctrl R
    (copy of sample list)
    for project folder
```

Figure 4 (con't)

	Test ID
GC/MS Analyses (Aqueous)	
CLP/HSL Semivolatiles	625-RAS-A
CLP/HSL Volatiles Library Search	624-RAS-A
GC/MS Analyses (Solids)	
CLP/HSL Semivolatiles	8270-RAS-S-S
CLP/HSL Volatiles	8240-RAS-S
GC Analyses (Aqueous)	
CLP/HSL Organochlorine Pesticides/PCB	608-RAS-A
GC Analyses (Solids)	
CLP/HSL Organochlorine Pesticides/PCB	8080-RAS-S-S
Organic Prep Analyses	
Percent Moisture*	Percent Water-OMS-S
pH*	pH-MET-RAS-S

* These tests do not apply to MS and MSD samples.

NOTE: Holding times for CLP projects are normally calculated from the received date.

Figure 5

Case/SAS No: _____ NO. & TYPE _____

Contract No: _____ Confirmed: _____

Controller: _____ Region: _____ DATE REC'D _____

Turnaround: _____ Price: _____

Parameters: Metals _____ CN _____ DATE DUE _____

Others: _____

_____

RAS Invoice No. _____ SAS Invoice No: _____

Figure 6

SAMPLE DELIVERY GROUP (SDG)
TRAFFIC REPORT (TR) COVER SHEET

Lab Name: Rocky Mountain Analytical Laboratory Contract No.: _____

Lab Code: ENSECO Case No.: _____ SAS No.: _____ - _____

Full Sample Analysis Price in Contract: $_____

SDG No./First Sample in SDG: _____ Sample Receipt Date: ___/___/___
(Lowest EPA Sample Number in first shipment of samples received under SDG) (MM/DD/YY)

Last Sample in SDG: _____ Sample Receipt Date: ___/___/___
(Highest EPA Sample Number in last shipment of samples received under SDG) (MM/DD/YY)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1	_____	11	_____
2	_____	12	_____
3	_____	13	_____
4	_____	14	_____
5	_____	15	_____
6	_____	16	_____
7	_____	17	_____
8	_____	18	_____
9	_____	19	_____
10	_____	20	_____

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

_____ _____
Sample Custodian Date

STANDARD
OPERATING
PROCEDURE

Subject or Title: SAMPLE STORAGE		Page 1 of 3
SOP No.: LP-RMA-5002	Revision No.: 1.0	Effective Date: 06/25/91
Supersedes: Original		

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1. Purpose:

   To effectively organize and maintain completed samples until they are either disposed of or returned to the client.

2. Policies:

   Sufficient refrigerator space to maintain unused EPA sample volume for 60 days after data submission and sample extracts for 365 days after data submission. Samples must be stored in an atmosphere demonstrated to be

Prepared by: QA Department	Date: 06/25/91
Management Approval: Tina Tyson	Date: 7-1-91
QA Officer Approval: Jan Rademacher	Date: 7-1-91

free from all potential contaminants. Volatile samples must be stored in a refrigerator used only for storage of volatile samples from this contract. Refer to the attached diagram for the storage locations for both organic and inorganic samples.

3. Safety:

Not applicable.

4. Procedure:

4.1 Check and document physical condition of sample.

4.2 Verify documentation and parameter assignment.

4.3 Log into LIMS (See SOP: LP-RMA-5001).

4.4 Send acknowledgement letter to client or SDG package.

4.5 Store sample according to preservation guidelines.

4.6 Transfer sample to lab with proper documentation.

4.7 Document analytical work.

4.8 Return unused sample to sample control.

4.9 Samples are kept in Archive area.

4.10 Return sample to client or arrange for sample disposal.

5. Responsibilities:

Sample custodian is responsible for maintaining and disposing of completed samples.

6. Definitions

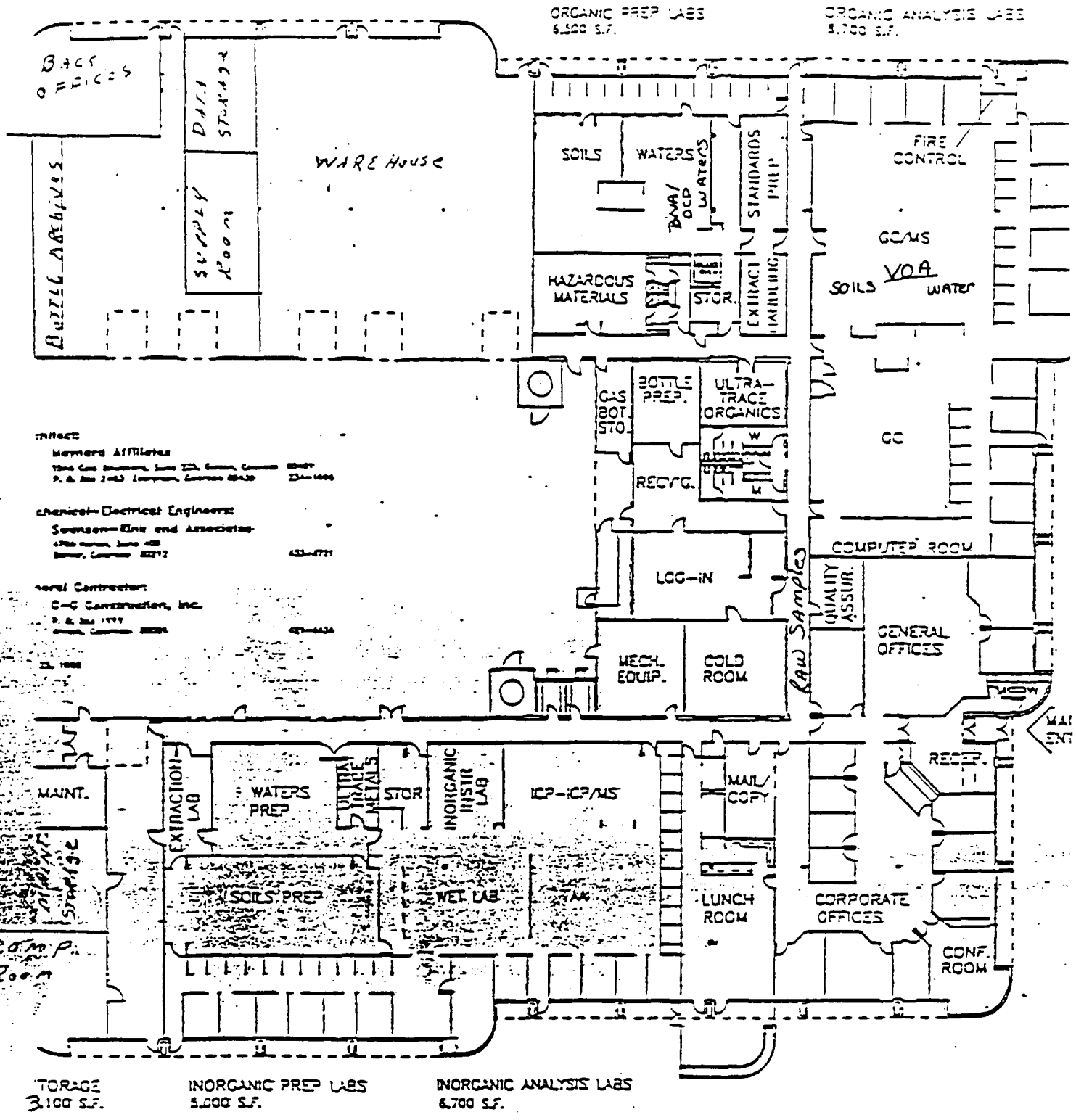
6.1 LIMS - Laboratory Information Management System, commercial and custom software programs designed to perform calculations, check results, generate reports, and ensure data integrity and security.

6.2 Sample - a portion of material to be analyzed that is contained in single or multiple containers and identified by a unique sample number.

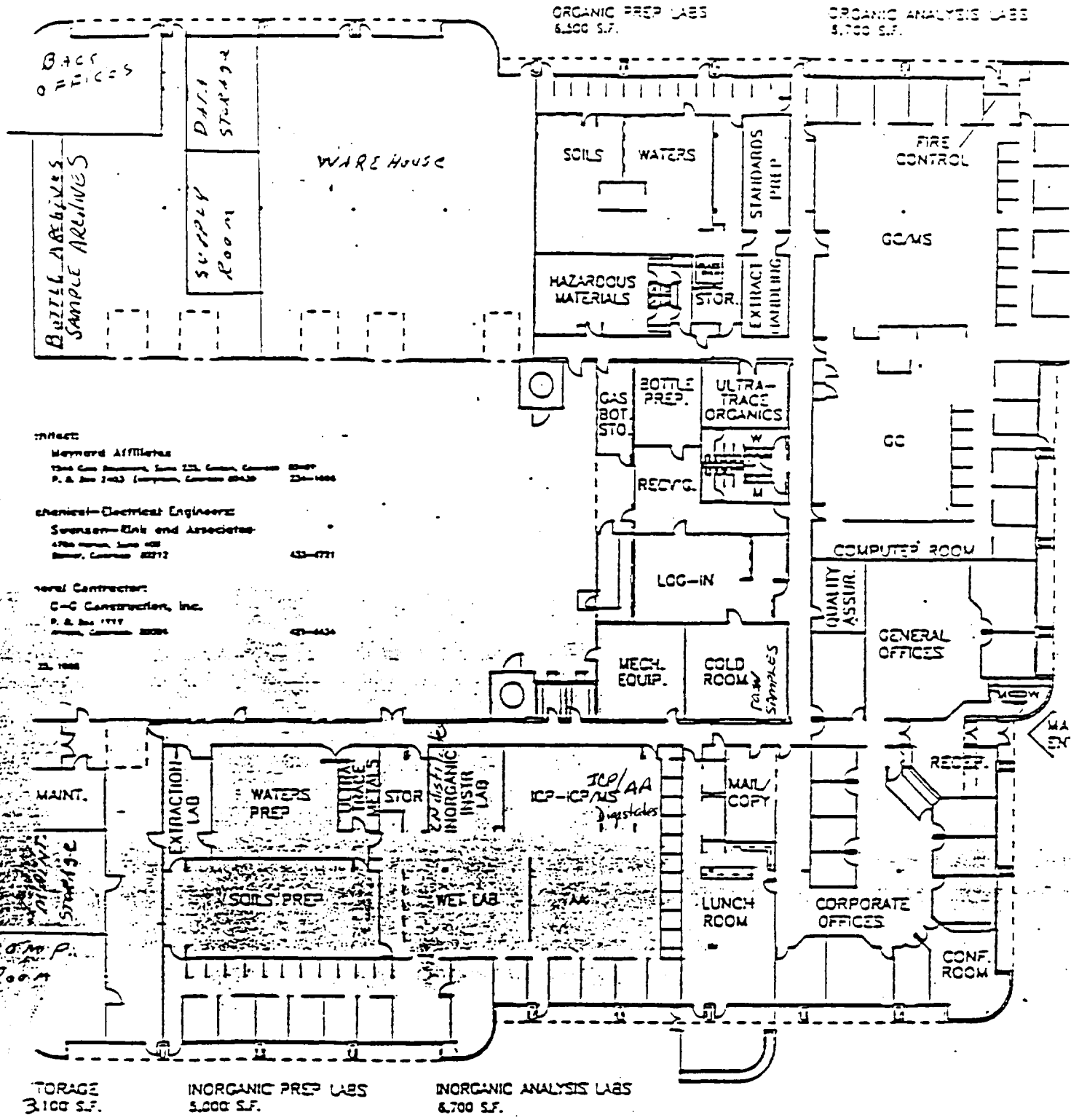
6.3 Sample Delivery Group (SDG) - a unit within a single Case that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a Case, received over a period of up to 14 calendar days. Data from all samples in an SDG are due concurrently. A Sample Delivery Group is defined by one of the following, which ever occurs first:

- Case; or
- Each 20 field samples within a Case; or
- Each 14-day calendar period during which field samples in a Case or SDG.

Samples may be assigned to Sample Delivery Groups by matrix (i.e., all soils in one SDG, all waters in another), at the discretion of the laboratory.



EPA Sample Storage Areas
For Organic Analyses



Rocky Mountain Analytical Laborator

A Division of ENSECO, Incorporat

EPA Sample Storage Area
for Inorganic Analysis

STANDARD
OPERATING
PROCEDURE

Subject or Title: BUILDING SECURITY		Page 1 of 3
SOP No.: LP-RMA-5003	Revision No.: Original	Effective Date: 09/28/90
Supersedes:		

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1. Purpose:

   The purpose of building security is to guarantee data security and confidentiality for the client as well as providing analytical data which is legally defensible.

2. Policies:

   RMAL's security policy includes controlled access to the building, testing areas and data files, confidentially agreements with all personnel, identification badges for all personnel, electronic security and fire alarm systems. All visitors are also assigned visitor badges and are accompanied by RMAL staff during their stay in the facility.

Prepared by: Sara Redenbarger	Date: 11/7/90
Management Approval: Gary Tok	Date: 11-12-90
QA Officer Approval: Sara Redenbarger	Date: 11/7/90

SOP No.:	Revision No.1	Effective Date:
LP-RMA-5003	Original	09/28/90

3. Safety Issues: Not Applicable

4. Procedure:

4.1 Building Security

4.1.1 All exterior doors to the facility will remain locked at all times with the exception of the front entrance.

4.1.2 During the hours of 7:00 a.m. to 6:00 p.m., the front entrance or main reception area is controlled by the receptionist and secured by locked entries. The alarm system is not activated during this time period.

4.1.3 The last employee to leave the facility must page the building to ensure he or she is the last person in the facility, they then call the alarm company to identify themself by name and code. The alarm system is activated, during this time period to prevent all other exterior doors from being useable, including sample receiving and the patio doors.

4.1.4 Sample receiving during the hours of 6:00 p.m.to 7:00 a.m. is permitted only with the assistance by a member of sample control.

4.2 Personnel Identification

4.2.1 All employees and visitors are required to wear security badges at all times while on the premises of all ENSECO divisions.

4.2.2 The personnel administrator is responsible for issuing a picture I.D. badge to an employee on the employee's first day of employment. Each employee is responsible for his/her badge. Additionally, each employee will be required to sign a "Confidentiality Agreement" which is included in the employee's personnel file.

4.2.3 The receptionist is responsible for issuing a badge to each visitor to the facility. Visitors must request a badge from the front office of the division they visit, sign the visitor log and must be accompanied by an ENSECO employee before access to any building will be allowed.

4.3 Building Alarm System

4.3.1 Each employee will receive a copy of the security manual, personal security code and security training at the time of their orientation provided by a member of the Human Resources department. The procedure is confidential information and can only be obtained from the Personnel Department.

5. Responsibilities:

5.1 It is the responsibility of each employee to maintain confidentiality of all clients data.

5.2 The Personnel Department is responsible for issuing employee identification badges and having signed "Confidentiality Agreements" in each employee's personnel file.

5.3 The receptionist is responsible for issuing visitor badges and for visitor sign-in during normal business hours.

5.4 Employees escorting visitors are responsible for ensuring that visitation procedures are followed and that data confidentiality has not been compromised.

6. Comments:

Enseco
CORNING Company

STANDARD
OPERATING
PROCEDURE

Subject or Title: Page 1 of 3

Bottle Preparation and Cooler Shipment

SOP No.:	Revision No.:	Effective Date:
LP-RMA-0056	1.0	July 23, 1990

Supersedes: Original

1. Purpose

   To assure that the proper procedures are followed when preparing sample bottles and coolers for shipment.

2. Policies

   The sample bottles are prepared according to ENSECO QAPP Revision 3.3. Coolers are shipped by either UPS or overnight courier requirements.

3. Safety Issues

   Due to the use of both base and acid as preservatives, safety glasses should be worn along with lab coats and gloves.

4. Procedure

   4.1 The client defines the scope of work to a Program Administrator and the PA completes a bottle order sheet which is given to the sample control technician (see example sheet #7.)

   4.2 The sample technician fills the bottle order using the appropriate bottles and preservatives as stated on the attached guideline sheet (see example sheet #1.) The bottles the laboratory provides for sampling and analysis are purchased from I-Chem. These bottles are provided with certificates of analysis and assigned QC lots (see example sheet #2.) The appropriate QC lots are noted on each bottle order sheet and on each glass bottle.

   4.3 The VOA trip blanks are labeled with the date, the sample technician's name and the words "trip blank."

Prepared by:	Date:
Bob Musil	July 23, 1990
Management Approval:	Date:
Steve Mark	July 25, 1990
QA Officer Approval:	Date:
Jan Zimmerman	7/25/90

4.4 The following paperwork is enclosed with the cooler(s):

4.4.1. Sample labels for each bottle sent

4.4.2 Chain-of-custody forms

4.4.3. Cooler labels to be used for shipping and returning cooler(s)

4.4.4 Cooler seals

4.4.5 Guideline sheet for sample bottles and preservatives

4.5 The sample bottles are securely packed in the correct number of shipping containers with styrofoam. The styrofoam sleeves are placed around the bottles and styrofoam sheets are layered on top of the bottles before taping the cooler(s) shut.

4.6 The cooler(s) are taped shut with strapping tape and labeled with recipient's address.

4.7 Cooler seals are placed over the lid of each cooler(s).

4.8 Prepare shipping bill according to the client's request, whether it be UPS or Federal Express, as follows:

4.8.1. Federal Express

4.8.1.1 An airbill must be completed with the sample control technician's name, date and telephone number along with the recipient's name, address and telephone number (see example sheet #3.)

4.8.1.2 Confirm whether the charges are going to bill sender or bill recipient's Federal Express number and check the appropriate box.

4.8.1.3 Check the appropriate box for the service requested e.g. priority 1, standard air, deliver weekday or Saturday delivery.

4.8.1.4 Annotate the number of coolers being shipped, the total weight and declared value, if any.

4.8.1.5 The multiple shipment labels (see example sheet #4) must be used if shipping more than one cooler. Document the number of coolers being shipped, the date, and the number of the master airbill which is located at the top of the original airbill.

4.8.1.6 Place one bar code label on each cooler, excluding the cooler which will be shipped with the master airbill. A copy of the the bar code list is placed on the space provided on the back of the master airbill.

4.8.2 UPS (see example sheet # 5)

4.8.2.1 Address UPS pickup record with the client's name and address.

4.8.2.2 A blank space is provided for the zone number on the pick up record. The zone number is determined by using the first three digits of the zip code e.g. 80000. Use the zone chart (see example sheet #6) to determine the UPS zone.

5. Responsibility

Bottle preparation and cooler shipment is the responsibility of the sample control technicians. Initiating the bottle order form(s) is the responsibility of the Program Administrator.

6. Comments

It is important to pay close attention to which preservative (acid or base) is being added to each type of bottle.

7. Definitions

None.

Example Sheet #1

Enseco
A CORNING Company

GUIDELINES FOR SAMPLE BOTTLES AND PRESERVATIVES

E: In addition to the preservatives stated below, all samples should be cooled to 4°C

Number	Parameters	Container	Preservatives
LIQUID:	Aqueous		
1	Alkalinity, BOD, Chloride, Color, Res. Chlorine, pH, Chromium (VI), Conductance, Fluoride, Nitrite, MBAS, Ortho-Phos., Solids, Sulfate, Sulfite, Turbidity	32oz poly (WM)	None
2	Ammonia, COD, Nitrate, TKN, TON Nitrate + Nitrite, Total Phos., TOC, Phenolics	16oz glass (BR)	2 mL 50% Sulfuric Acid
3	TPH, Oil & Grease	32oz glass (BR)	4 mL 50% Sulfuric Acid
4	Metals, Hardness	16oz poly (WM)	10 mL 20% Nitric Acid
5	Gross Alpha, Gross Beta, Uranium, Radium 226, Radium 228	Two 32oz poly (BR)	10 mL 20% Nitric Acid
6	Total and/or Free Cyanide	8oz poly (WM)	2 mL 50% Sodium Hydroxide
7	Sulfide	8oz poly (WM)	1 mL 1N Zinc Acetate + 1 mL 50% Sodium Hydroxide
8	Fecal or Total Coliform (use 2 bottles if both required)	4 1/2 oz. poly, sterilized	None
10	THM	Three 40 mL glass vials	100 uL Sodium Thiosulfate
11	VOA, Purgeable Organics	Three 40 mL glass vials	200 uL 50% Hydrochloric Acid
12	Base Neutral/Acid Compounds	Two 32oz glass(BR)	None
13	Pesticides, PCBs	Two 32oz glass(BR)	None
14	Herbicides	1 32oz glass(BR)	None
15	TOX--Single:	8oz glass (BR) (amber)	1 mL 50% Sulfuric Acid
	--Quad:	Four 8oz glass OR 1 32oz glass (amber)	1 mL 50% Sulfuric Acid 4 mL 50% Sulfuric Acid
20, 21	Bulk water analysis	1/2 Gal OR 1 Gal glass (WM)	None
OLID:	Soils, Sludges, Wastes		
30	Organics,TPH,Metals,RAD, Oil & Grease	16 oz glass (WM)	None
31	Wet Chem not listed for #30	8 oz glass (WM)	None
		4 oz glass (WM)	None

CHEM

CHEM RESEARCH

Example Sheet #2

CERTIFICATE OF ANALYSIS

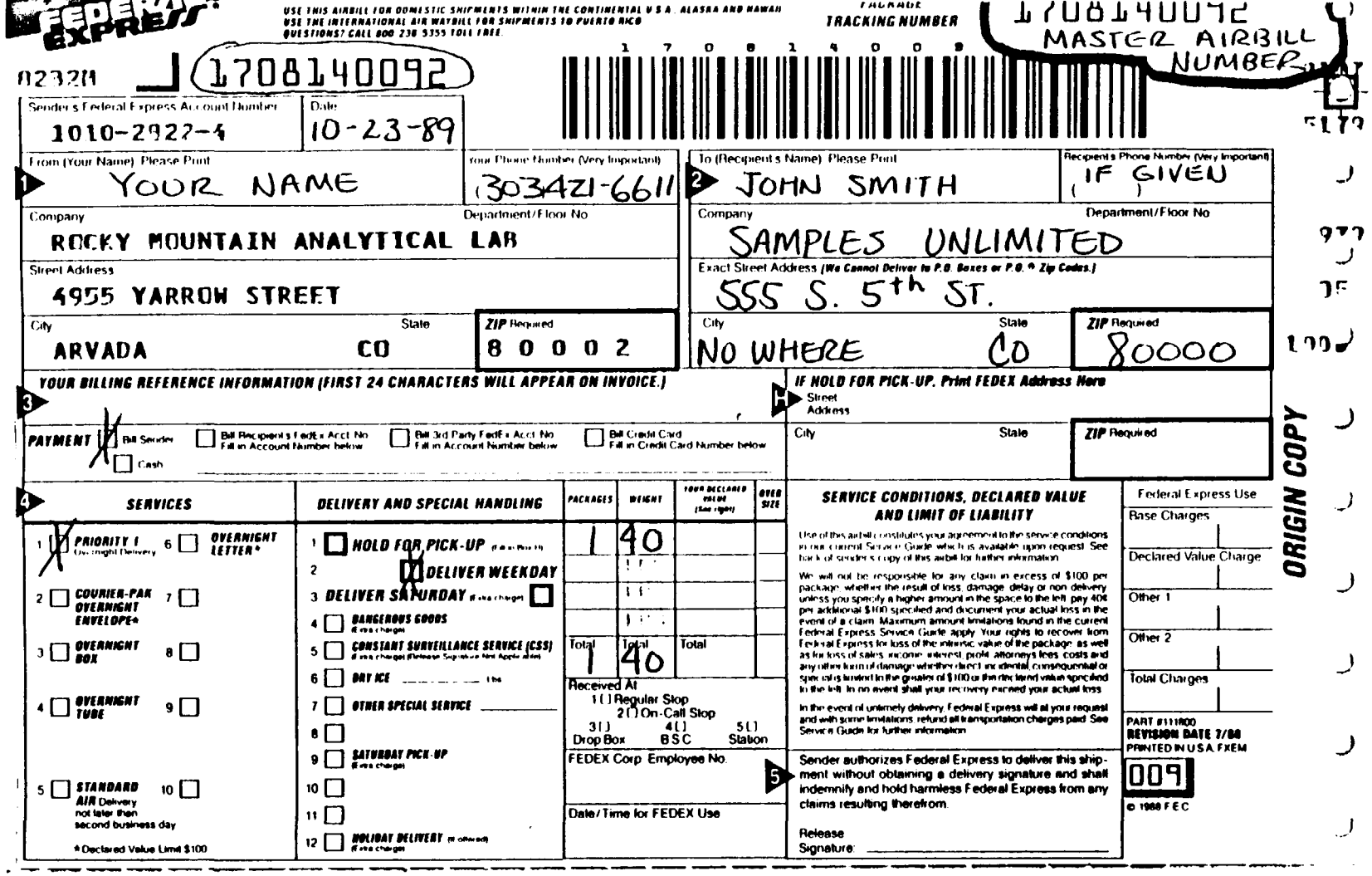
Analysis of Lot Number B9138CIT

The following compounds were analyzed for but not detected, or detected at a concentration of less than 5 micrograms/liter:

Chloromethane	Bromomethane	Vinyl Chloride
Chloroethane	Methylene Chloride	Acetone
Carbon Disulfide	1,1-Dichloroethene	1,1-Dichloroethane
Chloroform	1,2-Dichloroethane	trans-1,2-Dichloroethene
2-Butanone	1,1,1-Trichloroethane	Carbon tetrachloride
Vinyl acetate	Bromodichloromethane	1,2-Dichloropropane
Trichloroethene	Dibromochloromethane	1,1,2-Trichloroethane
Benzene	Bromoform	cis-1,3-Dichloropropene
2-Hexanone	4-Methyl-2-pentanone	Tetrachloroethene
Toluene	Chlorobenzene	Ethylbenzene
Styrene	Xylenes	1,2-Dichlorobenzene
1,3-Dichlorobenzene	1,4-Dichlorobenzene	trans-1,3-Dichloropropene
1,1,2,2-Tetrachloroethane		2-Chloroethyl vinyl ether

Please retain for your records and to aid in any correspondence with I-CHEM regarding lot B9138CIT.

Certified
Anita Rudd
President



FEDERAL EXPRESS

USE THIS AIRBILL FOR DOMESTIC SHIPMENTS WITHIN THE CONTINENTAL U.S.A., ALASKA AND HAWAII
USE THE INTERNATIONAL AIR WAYBILL FOR SHIPMENTS TO PUERTO RICO
QUESTIONS? CALL 800 238 5355 TOLL FREE.

PACKAGE TRACKING NUMBER 1708140092

MASTER AIRBILL NUMBER

IMPROVE COPY LEGIBILITY
PRESS HARD – PLEASE PRINT
USING A BALL POINT PEN

A23211 1708140092

Sender's Federal Express Account Number: 1010-2922-4
Date: 10-23-89

1 From (Your Name) Please Print: YOUR NAME
Your Phone Number (Very Important): (303)421-6611
Company: ROCKY MOUNTAIN ANALYTICAL LAB
Department/Floor No:
Street Address: 4955 YARROW STREET
City: ARVADA State: CO ZIP Required: 8 0 0 0 2

2 To (Recipient's Name) Please Print: JOHN SMITH
Recipient's Phone Number (Very Important): IF GIVEN ( )
Company: SAMPLES UNLIMITED
Department/Floor No:
Exact Street Address (We Cannot Deliver to P.O. Boxes or P.O. Zip Codes.): 555 S. 5th ST.
City: NO WHERE State: CO ZIP Required: 80000

3 YOUR BILLING REFERENCE INFORMATION (FIRST 24 CHARACTERS WILL APPEAR ON INVOICE.)

PAYMENT [X] Bill Sender [ ] Bill Recipient's FedEx Acct. No. Fill in Account Number below [ ] Bill 3rd Party FedEx Acct. No. Fill in Account Number below [ ] Bill Credit Card Fill in Credit Card Number below [ ] Cash

4 SERVICES

1 [X] PRIORITY 1 Overnight Delivery
2 [ ] COURIER-PAK OVERNIGHT ENVELOPE*
3 [ ] OVERNIGHT BOX
4 [ ] OVERNIGHT TUBE
5 [ ] STANDARD AIR Delivery not later than second business day
6 [ ] OVERNIGHT LETTER*
7 [ ]
8 [ ]
9 [ ]
10 [ ]
*Declared Value Limit $100

DELIVERY AND SPECIAL HANDLING

1 [ ] HOLD FOR PICK-UP
2 [X] DELIVER WEEKDAY
3 DELIVER SATURDAY [ ]
4 [ ] DANGEROUS GOODS
5 [ ] CONSTANT SURVEILLANCE SERVICE (CSS)
6 [ ] DRY ICE ____ lbs
7 [ ] OTHER SPECIAL SERVICE ____
8 [ ]
9 [ ] SATURDAY PICK-UP
10 [ ]
11 [ ]
12 [ ] HOLIDAY DELIVERY

PACKAGES	WEIGHT	YOUR DECLARED VALUE (See right)	OVER SIZE
1	40		
Total 1	Total 40	Total	

Received At
1 [ ] Regular Stop
2 [ ] On-Call Stop
3 [ ] Drop Box 4 [ ] BSC 5 [ ] Station

FEDEX Corp. Employee No.

Date/Time for FEDEX Use

IF HOLD FOR PICK-UP, Print FEDEX Address Here
Street Address
City State ZIP Required

SERVICE CONDITIONS, DECLARED VALUE AND LIMIT OF LIABILITY

Use of this airbill constitutes your agreement to the service conditions in the current Service Guide which is available upon request. See back of sender's copy of this airbill for further information.

We will not be responsible for any claim in excess of $100 per package, whether the result of loss, damage, delay or non-delivery unless you specify a higher amount in the space to the left, pay 40¢ per additional $100 specified and document your actual loss in the event of a claim. Maximum amount limitations found in the current Federal Express Service Guide apply. Your rights to recover from Federal Express for loss of the intrinsic value of the package, as well as for loss of sales, income, interest, profit, attorney's fees, costs and any other form of damage whether direct, incidental, consequential or special is limited to the greater of $100 or the declared value specified in the left. In no event shall your recovery exceed your actual loss.

In the event of untimely delivery, Federal Express will at your request and with some limitations, refund all transportation charges paid. See Service Guide for further information.

5 Sender authorizes Federal Express to deliver this shipment without obtaining a delivery signature and shall indemnify and hold harmless Federal Express from any claims resulting therefrom.

Release Signature: _____

Federal Express Use
Base Charges
Declared Value Charge
Other 1
Other 2
Total Charges

PART #111800
REVISION DATE 7/88
PRINTED IN U.S.A. FXEM
009
© 1988 FEC

ORIGIN COPY

1708140092 1708140092

EXAMPLE SHEET #3

DOMESTIC AND INTERNATIONAL MULTIPLE PACKAGE SHIPMENT LABELS

MULTIPLE PACKAGE SHIPMENT LABELS

SHIPMENT DATE	10-23-89
MASTER AIRBILL NUMBER	170 8140092

2 OF 4 — 403 6174 852

DESCRIPTION _____

3 OF 4 — 403 6174 861

DESCRIPTION _____

___ OF ___ — 403 6174 877

DESCRIPTION _____

___ OF ___ — 403 6174 886

DESCRIPTION _____

___ OF ___ — 403 6174 895

DESCRIPTION _____

PART #105972 WCSC REV. 10/88 • 1988 F.E.C.
SENDER'S COPY 8-89

FEDEX COURIER: These labels are designed to be used for multiple package shipments. This system should be used for all FedEx Express packages both Domestic and International.
Enter shipment date and master airbill number. Each package within the shipment should be sequentially numbered starting with Package Number 2 of ___ (Package Number 1 will have the master airbill affixed to it). The shipper may enter a description of the commodity if desired (required for international shipments). If all five (5) tracking numbers are not used, those numbers should be voided.

An address label reflecting exact recipient address, must be affixed to each package within a multiple package shipment. Place the entire 7" label that includes the barcode and the customer package tracking number directly above the recipient address label on each package within the shipment except the package which has the original airbill attached.
Destroy any unused labels.

- **After completion, the MPS Sender copy (pink) is given to the sender.**
- **The self adhesive MPS copy is attached to the back of the Recipient copy of the master airbill for Domestic shipments. Attach it to *the back of the Hub Manifest copy for International shipments originating* in the U.S. and to the back of the Origin Accounting copy for shipments originating outside the U.S., Hawaii and Alaska.**

403 6174 852

403 6174 861

403 6174 877

403 6174 886

403 6174 895

MPS 403 6174 852
FEDERAL EXPRESS 403 6174 852
CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

MPS 403 6174 861
FEDERAL EXPRESS 403 6174 861
CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

MPS 403 6174 877
FEDERAL EXPRESS 403 6174 877
CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

MPS 403 6174 886
FEDERAL EXPRESS 403 6174 886
CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

MPS 403 6174 895
FEDERAL EXPRESS 403 6174 895
CUSTOMER PACKAGE TRACKING NUMBER — PULL UP PURPLE TAB

EXAMPLE SHEET #4

United Parcel Service

PICKUP RECORD

DATE / /

REC'D FROM

PICKUP RECORD NO. 081778289

CHECK CODE 5

STAMP YOUR UPS SHIPPER NUMB

LBS CL 810-5

ENTER EACH PACKAGE ON A SEPARATE LINE. IF RECORD IS VOIDED, PLEASE GIVE TO DRIVER.
INCREASE FRACTIONS OF A POUND TO NEXT FULL POUND.

REFERENCE NO	NAME — STREET — CITY	STATE	ZIP CODE	TYPE SERVICE: GROUND ZONE In State	GROUND ZONE Out of State	GROUND LBS	AIR ZONE	AIR LBS	(✓) OS*	DECLARED VALUE** IF IN EXCESS OF $100 00	COD*** AMOUNT
1	SPARTON TECH 9621 COORS Rd. N.W. ALBUQUERQUE	NM	87114		4	4					
2	EAST 2200 COTTONTAIL LANE SOMERSET	NJ	08873		7	2					
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											

EXAMPLE SHEET #5

ED BY	PICKUP TIME	NO PKGS	NO CALLS

* OS (Oversize) applies if less than 25 lbs. and more than 84 inches in length and girth combined.
** Unless a greater value is declared in writing on this receipt, the shipper hereby declares and agrees that the released value of each package or article not enclosed in a package covered by this r $100, which is a reasonable value under the circumstances surrounding the transportation. The rules relating to liability established by the Warsaw Convention and any amendments thereto shall the international carriage of any shipment hereunder insofar as the same is governed thereby. The entry of a C.O.D. amount is not a declaration of value. In addition, the maximum value for an ai package is $25,000 and the maximum carrier liability is $25,000. Claims not made to carrier within 9 months of the scheduled delivery date are waived.
*** All checks in payment of C.O.D.'s accepted at shipper's risk.

6-88 (600M BKS. 12-88)U

EXAMPLE SHEET #6

GROUND SERVICE ZONE CHART

For Shippers with ZIP Codes 800-01 to 802-99

Service to 48 Continental United States

To determine zone, take first three digits of ZIP Code to which parcel is addressed and refer to chart below.

ZIP CODE PREFIXES	UPS ZONE
004-005	7
010-043	7
044	8
045	7
046-048	8
049-089	7
100-139	7
140-147	6
148-149	7
150-167	6
168-199	7
200-214	7
215	6
216-225	7
226	6
227	7
228-229	6
230-239	7
240-253	6
254	7
255-274	6
275-279	7
280-282	6
283-285	7
286-293	6
294-295	7
296-299	6
300-319	6
320-322	7
323-325	6
326-339	7
342-349	7
350-369	6
370-372	5
373-374	6
375	5
376-379	6
380-384	5
385	6
386-392	5
393-395	6
396-397	5
399	6
400-418	6
420	5
421-422	6
423-424	5
425-459	6
460-466	5
467-468	6
469	5
470-471	6
472	5
473	6
474-479	5
480-489	6
490-491	5
492	6
493-495	5
496-497	6
498-499	5
500-503	4
504	5
505	4
506-507	5
508-516	4
520-560	5
561-562	4
563-567	5
570-576	4
577	3
580-583	5
584-593	4
594-595	5
596-597	4
598-599	5
600-639	5
640-649	4
650-659	5
660-675	4
676-679	3
680-689	4
690-693	3
700-704	6
705-729	5
730-732	4
733	5
734-738	4
739	3
740-746	4
747	5
748	4
749-761	5
762-763	4
764-789	5
790-799	4
800-810	2
811	3
812	2
813-815	3
816-820	2
821	4
822-823	3
824	4
825-827	3
828	4
829-830	3
831-834	4
835-838	5
840-853	4
854	5
855-873	4
874-877	3
878-883	4
884	3
885	4
889-892	5
893	4
894-897	5
898	4
900-954	5
955	6
956-961	5
970-979	5
980-982	6
983-984	5
985	6
986	5
988-994	5

See separate charts for UPS Next Day Air, 2nd Day Air and International services.
Air service is provided to all points in Alaska, Hawaii and Puerto Rico.

017232 REV. 2-

Example Sheet #7

BOTTLE ORDER

RMAL Client ID:_____ Request filled by:_____

Request By:_____ Date:_____ Time:_____

Coolers Billed To (specify Client ID):_____

Blue Ice Required: YES NO
Special Requirements:_____

DI____Gallons Carbon Free____Gallons Milli-Q____Gallons

No. of bottles	Standard Water	No. of Bottles	Bulk Water
_____	1. 32oz poly (WM)	_____	20. 1/2 gallon glass
_____	2. 16oz glass (BR), 50% H2SO4	_____	21. 1 gallon glass
_____	3. 32oz glass (BR), 50% H2SO4		
_____	4. 16oz poly (WM), 20% HNO3		
_____	5. 2-32oz poly (BR), 20% HNO3		Solids
_____	6. 8oz poly (WM), 50% NaOH		
_____	7. 8oz poly (WM), Zn Ac + NaOH	_____	30. 16oz glass (WM)
_____	8. 4.5oz poly, sterilize	_____	31. 8oz glass (WM)
_____	10. 3-40mL glass w/septa,Na2S2O3	_____	32. 4oz glass (WM)
_____	10A. Trip Blank		
_____	11. 3-40mL glass w/septa,HCL		
_____	11A. Trip blank		
_____	12. 2-32oz glass (BR)		
_____	13. 2-32oz glass (BR)		
_____	14. 32oz glass (BR)		
_____	15. Single: 8oz amber glass (BR)		
	Quad: 32oz amber glass (BR)		
	50% H2SO4		

Ship To:

_____

_____

_____

_____

_____

Client P.O.#_____

Pick-up on:__/__/__Time:_____

Delivered By (Date):__/__/__

Ship By:_____

_____

UPS Charges:_____

Fed X Charges:_____

Client Fed X Account #:_____

Invoice #:_____

Sample Safe / Cooler numbers:_____

Bottle lot #'s by type:_____

_____

STANDARD
OPERATING
PROCEDURE

Subject or Title:
SAMPLE RECEIPT AND CHAIN OF CUSTODY

SOP No.:	Revision No.:	Effective Date:
LP-RMA-0005	Original	12/9/87

Supersedes:

1. Purpose:

To document receipt of all samples to the laboratory. To notify lab personnel of all incoming samples. To notify lab personnel of arriving samples that contain short holding parameters. To record the transfer of samples from the client to the lab.

2. Policies:

Always assign a project number to every group of samples that arrive at the lab regardless of whether work is proceeded on them or not.

Project numbers are assigned in numerical order. USGS and MKE samples receive separate series of numbers. MKE samples require special chain of custody tracking.

3. Safety:

Always wear gloves and glasses while unpacking coolers. Coolers containing strong smelling samples must be unpacked under the hood area.

4. Procedure:

a. As samples arrive they are given a unique project number for each group of samples from one client and recorded in the log book (Figure 1).
b. Fill out the Sample checklist (Figure 2) while unpacking the samples.
c. For samples arriving by a courier check that the custody seals are intact.
d. Open the coolers, unpack the samples and check the information written on the chain of custody against what was received. Note any discrepancies such as missing samples, or broken bottles on the chain of custody form.
e. Label all the samples (usually by sampling sites) with a project number and unique sample number (1,2,3,etc.). Record these numbers on the chain of custody next to the client identifications.

Prepared by:	Date: 12/10/87
Management Approval:	Date: 12/10/87
QA Officer Approval: Robert C. Hamisch	Date: 12/9/87

f. Sign and date the Chain of Custody (Figure 3). For samples hand delivered have the client sign and relinquish the custody. Always retain the top copy with the samples and only give a bottom copy to the client.
g. Look for any inorganic short holding parameters and sign in these samples on the inorganic short holding clipboard (Figure 4). Look for any volatile parameters and sign these samples in on the Volatile clipboard (Figure 5).
h. Take a picture of the samples. Label a manila file folder with the project number. Place the picture, checklist, chain of custody and any paperwork received in the folder.
i. Deliver the file folder to the appropriate project manager.
j. Place the samples in boxes and store in the walk in cooler on special shelves pending log in. Bottles needed to analyze the short holding parameters are placed in a special location in the walk in cooler.

5. Responsibilities:

Sample receiving personnel are responsible for signing the chain of custody upon receipt of samples, for knowing the location of the samples except when used by an analyst, and for signing out maximum security samples. Sample receiving personnel are responsible for noting the short holding parameters only when indicated on the paperwork from the client. Client managers must notify sample receiving if others are to be included.

6. Comments:

For maximum security of samples (beyond the storage in the secured facility) an internal chain of custody is provided. Analysts must sign for the samples in a special book and sign them in on return. The samples are stored in one of 3 locked refrigerators.

PROJECT #	CLIENT NAME	PROJECT NAME/ #	# of COOLER	PARAMETERS/COMMENTS	DATE REC'D.	TIME REC'D	MATRX

FIGURE 2

SAMPLE CHECKLIST

PROJECT # | | | | | | DIV: | | | |

COMPANY NAME: _____

SAMPLE CHECKLIST DONE BY: _____

COOLER(S) # _____ (RMAL/CLIENT)	Y	N
PICTURE TAKEN:		
SEALS INTACT:		
COOLER TEMP OK:		
BOTTLES BROKEN OR LEAKING:		
CONTAINERS LABELED:		
RADIATION DETECTION:		
CHAIN OF CUSTODY:		
CC AGREES WITH SAMPLES:		
VOA SAMPLES FILLED COMPLETLY:		
SEDIMENT PRESENT IN WATERS:		
SAMPLE CORRECTLY PRESERVED:		
SHORT HOLDING TIMES:		

( )MS ( )VOA ( )60Z ( )IN

SAMPLE MATRIX: ( )WATER ( )SOIL ( )WASTE

OTHER: _____

TYPE OF BOTTLES: ( )RMA ( )CLIENT

DISCREPANCES: _____

Date_____

PROJECT NUMBER :								
SAMPLE NUMBERS :								
COLL. DATE/ TIME :								
REC. DATE/ TIME :								
PUT ON LIST BY :								
MASS/SPEC-VOA :								
G/C-VOA :								
GO2								
BTEX								
TCLP								
UNKNOWN :								
ANALYST :								
# OF VIALS/SAMPLE:								
COMMENTS :								

INORGANIC SHORT HOLDING TIME (≤ 48 hrs) AMETERS RECEIVED _____

Project Number																			
Sample Numbers																			
Client																			
Date Collected																			
Special Instructions																			
Logged By / Time																			
Approved By (CSR)																			
ASAP	Dissolved Oxygen(O_2)																		
	Sulfite ($SO_3$2-)																		
24 HOURS	pH (S)																		
	pH (Quad)																		
	Conductance (S)																		
	Conductance (Quad)																		
	Alkalinity																		
	Chlorine, Residual																		
	Chromium, Hexavalent (Cr^{+6})																		
	Coliform, Fecal																		
	Coliform, Total																		
	Odor																		
48 HOURS	BOD																		
	Color																		
	MBAS (Surfactants)																		
	Nitrate (NO^{3-})																		
	Nitrite (NO_2-)																		
	ortho-Phosphate																		
	Turbidity																		
	Settleable Solids																		
OTHER	TDS (Need Conductance (S))																		
	Ion Balance (Need pH (S), Conductance (S), Alkalinity)																		

Enseco - Rocky Mountain Analytical

4955 Yarrow Street
Arvada, Colorado 80002
303/421-6611 Facsimile: 303/431-7171

Attn: _____

CHAIN OF CUSTODY

No. 4999

Enseco Client _____

Project _____

Sampling Co. _____

Sampling Site _____

Team Leader _____

SAMPLE SAFE™ CONDITIONS

1. Packed by: _____ Seal # _____
2. Seal Intact Upon Receipt by Sampling Co.: Yes No
3. Condition of Contents: _____
4. Sealed for Shipping by: _____
5. Initial Contents Temp.: _____ °C Seal # _____
6. Sampling Status: Done Continuing Until _____
7. Seal Intact Upon Receipt by Laboratory: Yes No
8. Contents Temperature Upon Receipt by Lab: _____ °C
9. Condition of Contents: _____

Date	Time	Sample ID/Description	Sample Type	No. Containers	Analysis Parameters	Remarks

CUSTODY TRANSFERS PRIOR TO SHIPPING

	Relinquished by: (signed)	Received by: (signed)	Date	Time
1				
2				
3				

SHIPPING DETAILS

Delivered to Shipper by: _____

Method of Shipment: _____ Airbill # _____

Received for Lab: _____ Signed: _____ Date/Time _____

Enseco Project No. _____

White and Pink Copies to Lab Yellow to Sampler

SS-001

10. DATA REDUCTION, VALIDATION, AND REPORTING

Data Reduction and Validation

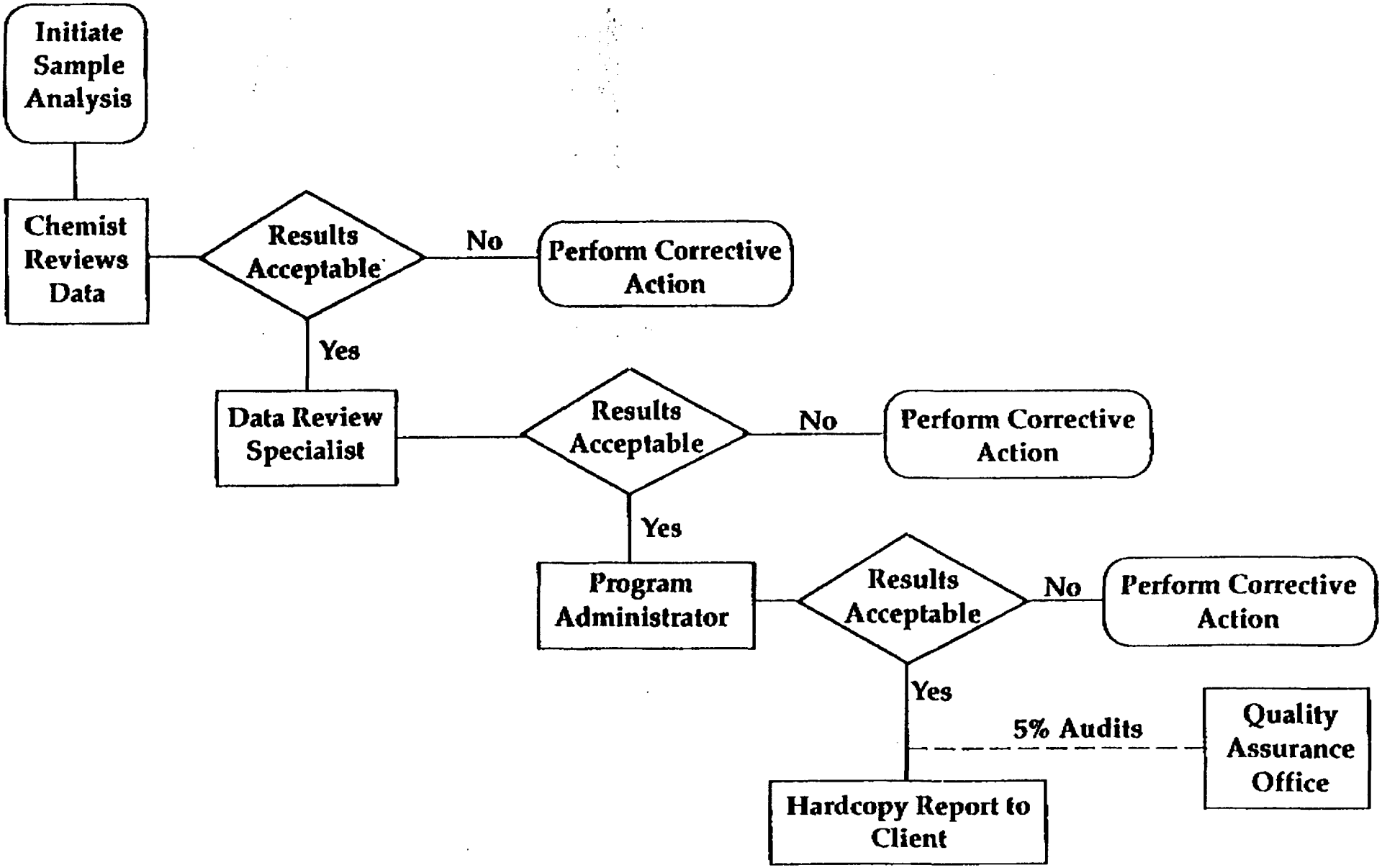
All analytical data generated within Enseco laboratories are extensively reviewed prior to report generation to assure the validity of the reported data. The data validation process consists of data generation, reduction, and three levels of documented review, as described below (also see Figure 10-1). In each stage, the review process is documented by the signature of the reviewer and the date reviewed.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data are generated and reduced following protocols specified in laboratory SOPs. Each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- Analytical results are correct and complete;
- QC samples are within established control limits;
- Blanks are within appropriate QC limits;
- Special sample preparation and analytical requirements have been met; and
- Documentation is complete (e.g., all anomalies in the preparation and analysis have been documented, anomaly forms are complete; holding times are documented, etc.).

Figure 10-1

Data Validation Scheme



The data reduction and validation steps are documented, signed and dated by the analyst. This initial review step, performed by the analyst, is designated Level 1 review. The analyst then passes the data package to an independent reviewer, who performs a Level 2 review.

Level 2 review is performed by a supervisor or data review specialist whose function is to provide an independent review of the data package. This review is also conducted according to an established set of guidelines and is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC samples are within established guidelines;
- Qualitative identification of sample components is correct;
- Quantitative results are correct;
- Documentation is complete and correct (e.g., anomalies in the preparation and analysis have been documented; anomaly forms are complete; holding times are documented, etc.);
- The data are ready for incorporation into the final report; and
- The data package is complete and ready for data archive.

Level 2 review is structured so that all calibration data and QC sample results are reviewed and all of the analytical results from 10% of the samples are checked back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety.

An important element of Level 2 review is the documentation of any errors that have been identified and corrected during the review

process. Enseco believes that the data package submitted by the analyst for Level 2 review should be free of errors. Errors that are found are documented and transmitted to the appropriate supervisor. The cause of the errors is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench.

Level 2 data review is also documented and the signature of the reviewer and the date of review recorded. The reviewed data are then approved for release and a final report is prepared.

Before the report is released to the client, the data are reviewed for completeness and to ensure that the data meet the overall objectives of the project. This review is labeled Level 3 review and is typically done by the Program Administrator.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality are generated consistently.

In addition to the three levels of review discussed above, the Divisional QA department randomly audits 5% of all projects reported. The QA audit includes verifying that holding times have been met, calibration checks are adequate, qualitative and quantitative results are correct, documentation is complete, and QC results are complete and accurate. During the review, the QA department checks the data from 20% of the samples back to the bench sheet. If no problems are found with the data package, the review is complete. If any problems are found with the data package, an additional 10% of the samples are checked to the bench sheet. The process continues until no errors are found or until the data package has been reviewed in its entirety.

Data Reporting

A variety of reporting formats, from computerized data tables, to complex reports discussing regulatory issues, to a CLP-deliverables package, are available. In general, Enseco reports contain:

General Discussion: Description of sample types, tests performed, any problems encountered and general comments are given.

Analytical Data: Data are reported by sample or by test. Pertinent information including dates sampled, received, prepared, and extracted are included on each results page. The Enseco reporting limit for each analyte is also given.

Laboratory Performance QC Information: The results (Percent Recovery and Relative Percent Difference) of the Laboratory Control Samples analyzed with the project are listed, together with the control limits. Also, the analytical results for method blanks generated during analysis of organic and metals parameters are given.

Matrix-Specific QC Information: Results of any sample duplicates, matrix spikes, matrix spike duplicates or other project-specific QC requested by the client are also reported.

Methodology: Reference for analytical methodology used is cited.

Custom Services: Special services including data interpretation, special consultation, and raw data packages (when requested) are included.

II. INTERNAL QC CHECKS

The Enseco QA/QC program monitors data quality with internal QC checks. Internal QC checks are used to answer two questions:

1) Are laboratory operations "in control," (i.e., operating within acceptable QC guidelines), during data generation?

2) What effect does the sample matrix have on the data being generated?

The first question is answered by Laboratory Performance QC. Laboratory performance QC is based on the use of a standard, control matrix to generate precision and accuracy data that are compared, on a daily basis, to control limits. This information, in conjunction with method blank data, is used to assess daily laboratory performance.

The second question is addressed with Matrix-Specific QC. Matrix-Specific QC is based on the use of an actual environmental sample for precision and accuracy determinations and commonly relies on the analysis of matrix spikes, matrix duplicates, and matrix spike duplicates. This information, supplemented with field blank results, is used to assess the effect of the matrix and field conditions on analytical data.

Laboratory Performance QC is provided as a standard part of every routine Enseco analysis. Matrix-Specific QC is available as an option to the client and should be specified based on the types of matrices to be analyzed and the Data Quality Objectives (DQOs) and regulatory requirements of the project. A complete discussion of the Enseco Internal QC Check program follows.

Laboratory Performance QC Program

Laboratory Performance QC is performed for every routine Enseco analysis to demonstrate that laboratory operations are "in control". The main elements of Laboratory Performance QC are:

APPENDIX D

GENERAL STANDARD OPERATING PROCEDURE FOR COMPUCHEM LABORATORIES, INC.

3.0 Quality Assurance Policy Statement

The Management of CompuChem® Laboratories is fully and firmly committed to the quality assurance program described in this QA Plan. Each director, manager and supervisor as well as their staff members, as assigned in accordance with this plan, is obligated to comply with its stated requirements, responsibilities and objectives.

The primary QA objective is to develop and implement procedures for sample receiving, chain-of-custody, sample preparation, laboratory analysis, data validation, and reporting that will provide data that are legally defensible in a court of law. Key aspects of these procedures are described in this QA Plan, while specific details are included in the laboratory's SOPs and QA SOPs -- all three references serving to document the elements of CompuChem's QA Program.

The QA Program will be maintained and expanded or modified as necessary, to ensure all reported data are of uncompromising quality. In order to determine whether QA objectives are met, sufficient quality control will be generated to evaluate precision, accuracy and completeness, and when possible, a statement regarding representativeness and comparability will be provided.

4.1 Quality Assurance Management

4.1 Introduction

With over 400 employees, CompuChem® offers the scientific and technical expertise needed to service the analytical and informational needs of our customers. In addition to our experienced analytical laboratory personnel (with specialized skills in organic and inorganic analyses) CompuChem® utilizes a computer systems staff that plans, develops, and implements software systems for data management and sample scheduling and control. To ensure that the analytical needs of our clients are met, customer service representatives are assigned to each account, providing a liaison between the customer and the laboratory.

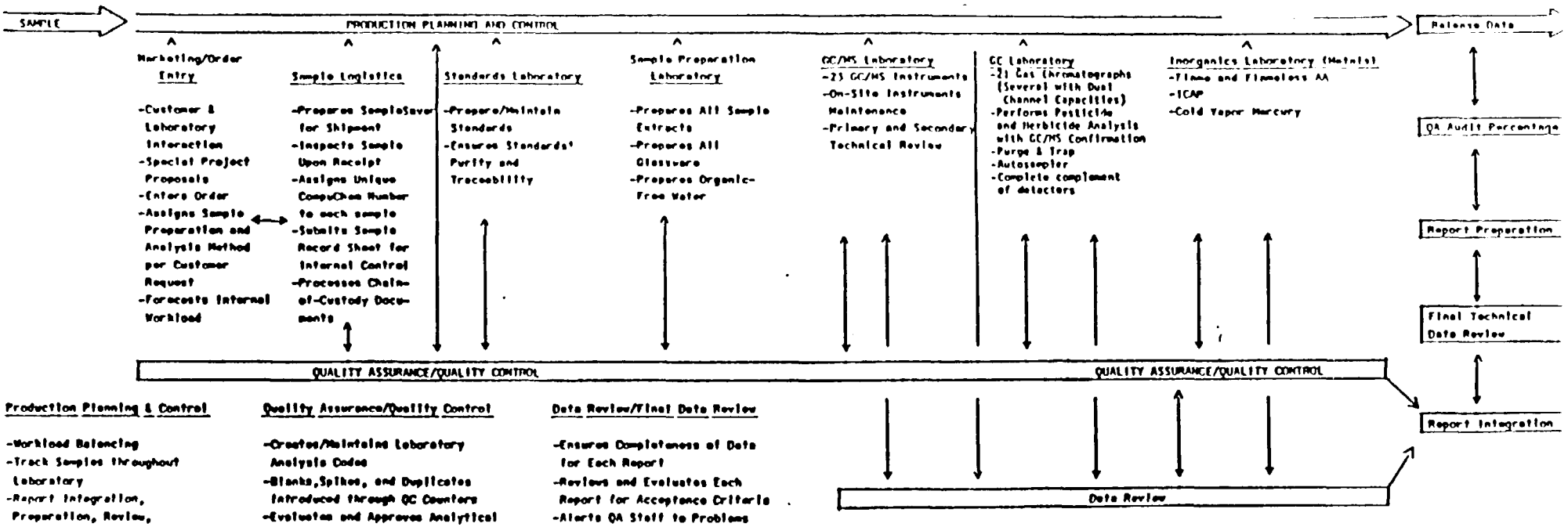
For our involvement with the EPA, dialogue has been established and is maintained with the Program Office in Washington, DC through our Project Officer and with our Deputy Project Officer located in our region (Region IV).

The following section describes the operational and functional responsibilities of key lab personnel, including the duties and services performed as they relate to product quality. Additionally, the roles and responsibilities of the Quality Assurance Department and its organizational relationship to lab management are identified. Please refer to the organizational charts at the end of this section for an overview of these relationships.

The Quality Assurance staff monitors and reviews all laboratory units and operates independently of production areas. All quality control criteria are documented and compliance is verified at each level of laboratory data review.

In-lab data validation and independent QA auditing SOPs describe the details of these quality control functions. Section 8.2 presents a general overview of the data review/data validation processes. The QA Department is responsible for, among other things, verifying the integrity of these functions and documenting performance for lab management review.

Laboratory Operations/Quality Assurance Relationships



Production Planning & Control

- -Workload Balancing
- -Track Samples throughout Laboratory
- -Report Integration, Preparation, Review, and Delivery
- -Monitors Analyses Due Dates
- -Customer Inquiry

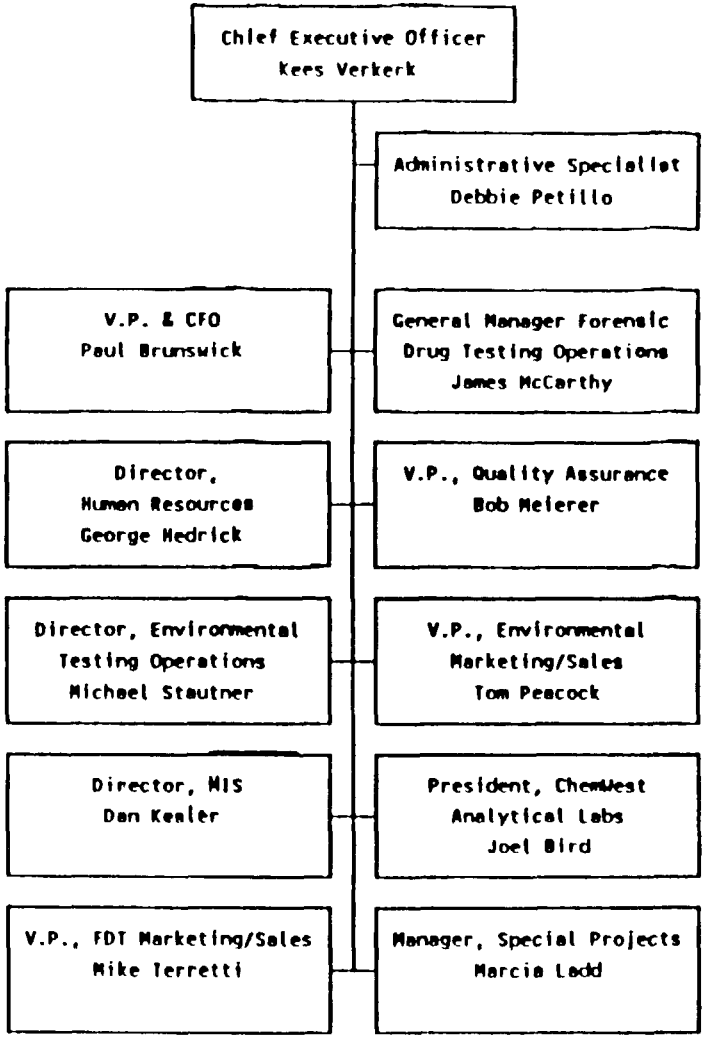
Quality Assurance/Quality Control

- -Creates/Maintains Laboratory Analysis Codes
- -Blanks, Spikes, and Duplicates introduced through QC Counters
- -Evaluates and Approves Analytical Standards
- -Monitors Surrogate Data Recovery Monitors Spike and Duplicate Results
- -Audits Data from All Laboratory Stations
- -Maintains Data Base of QC Data Produces QC Summary Reports for Feedback to Management

Data Review/Final Data Review

- -Ensures Completeness of Data for Each Report
- -Reviews and Evaluates Each Report for Acceptance Criteria
- -Alerts QA Staff to Problems
- -Verifies All Hits
- -Notes Deviations in Surrogate Recoveries

CompuChem Corporation
May 1, 1989



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Environmental Testing Operations
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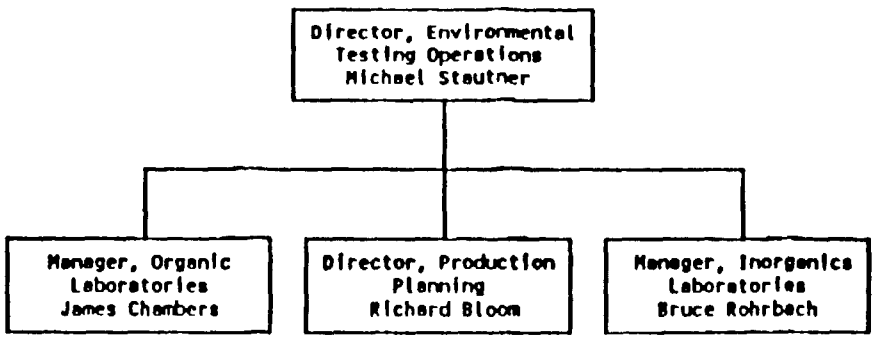
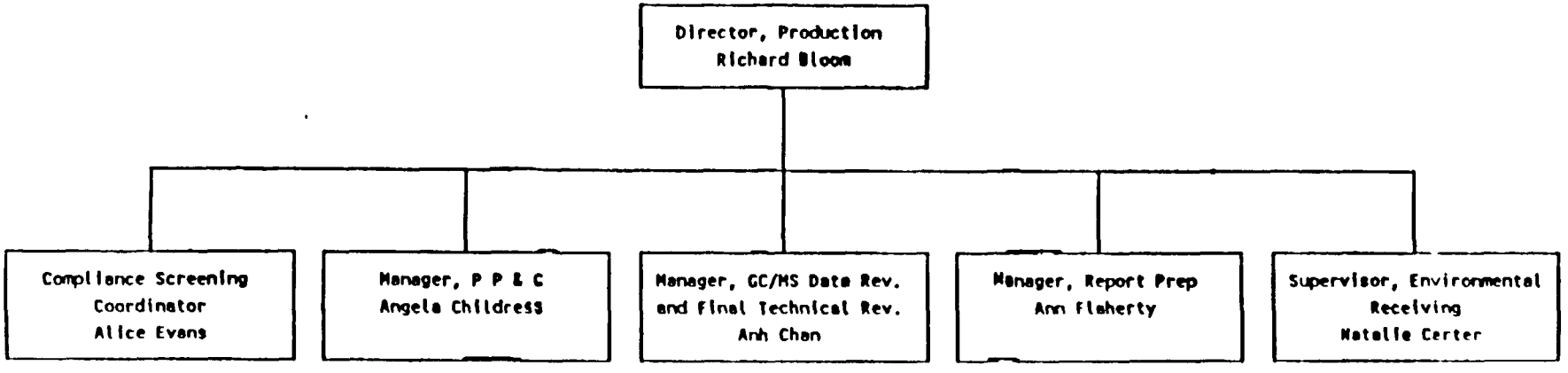
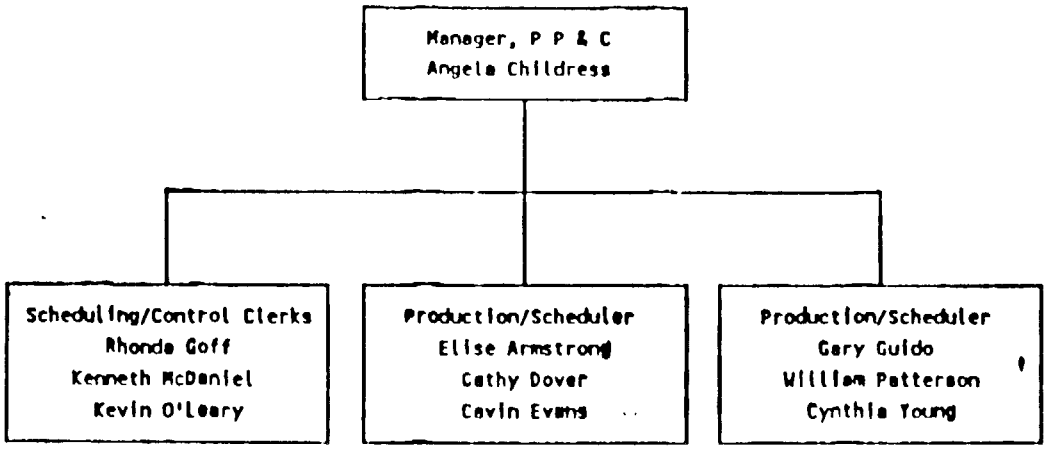


CompuChem Laboratories, Inc.
Production
May 1, 1989



CompuChem Laboratories, Inc.
P P & C
May 1, 1989



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Environmental Receiving
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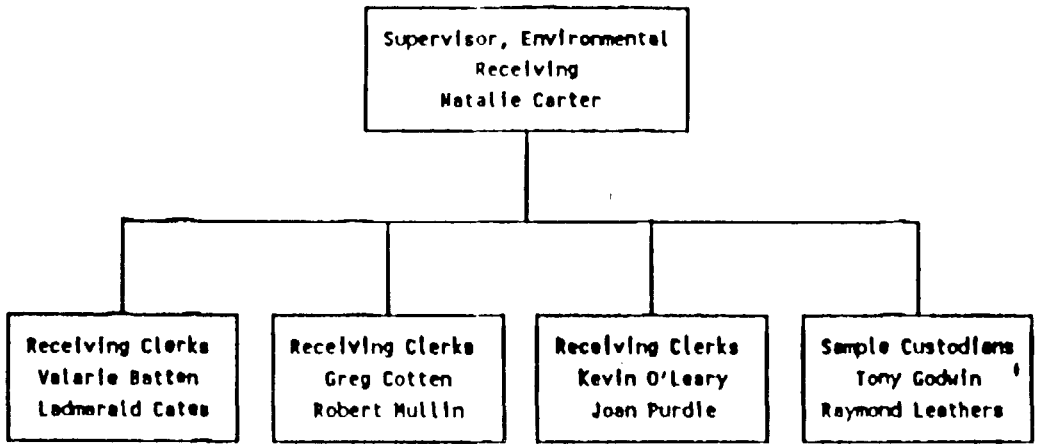
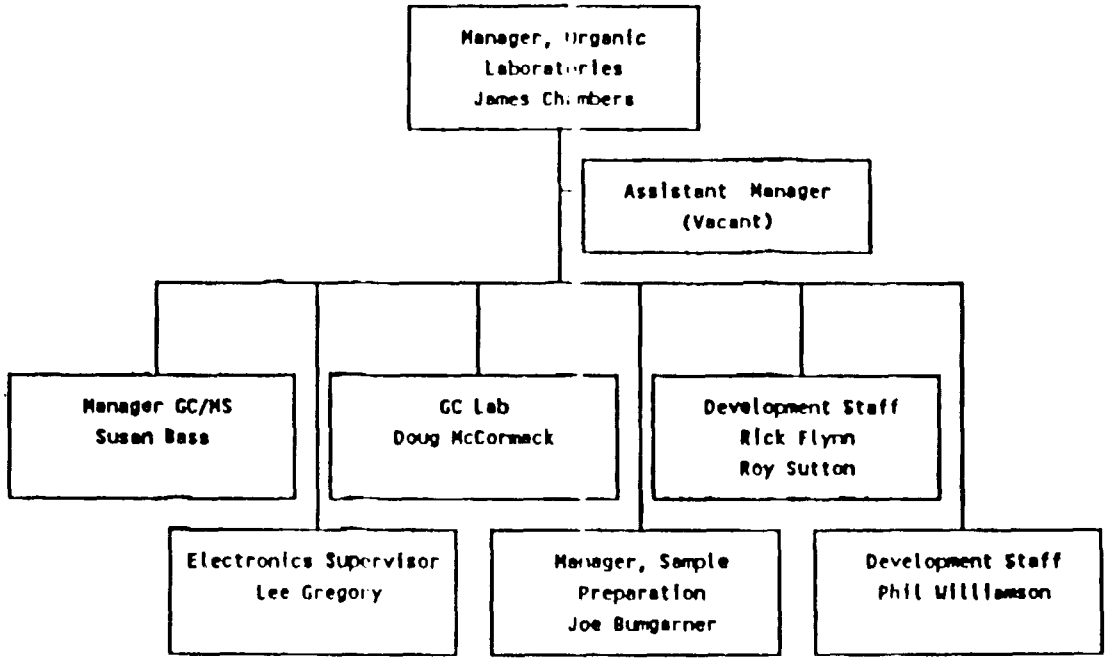
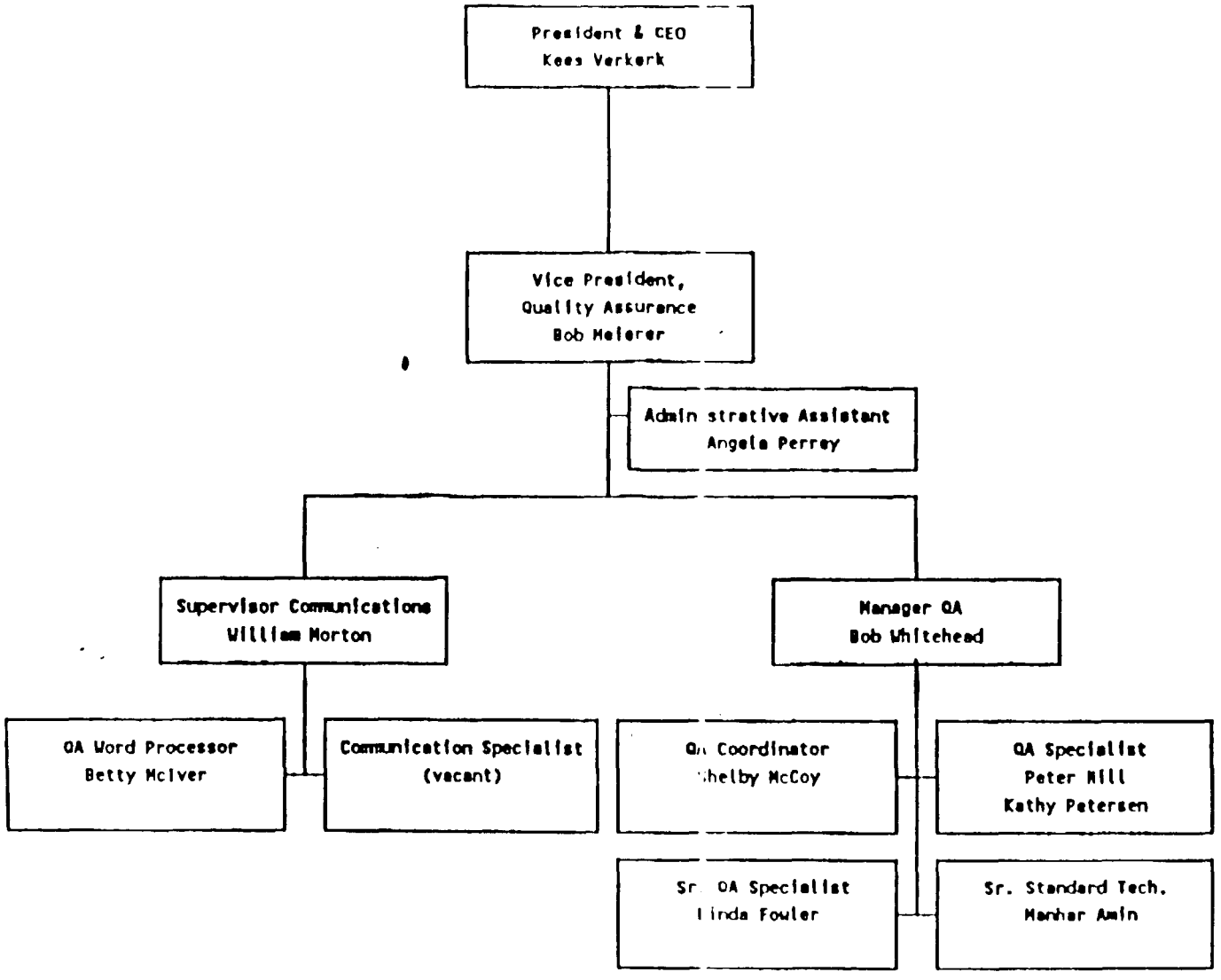
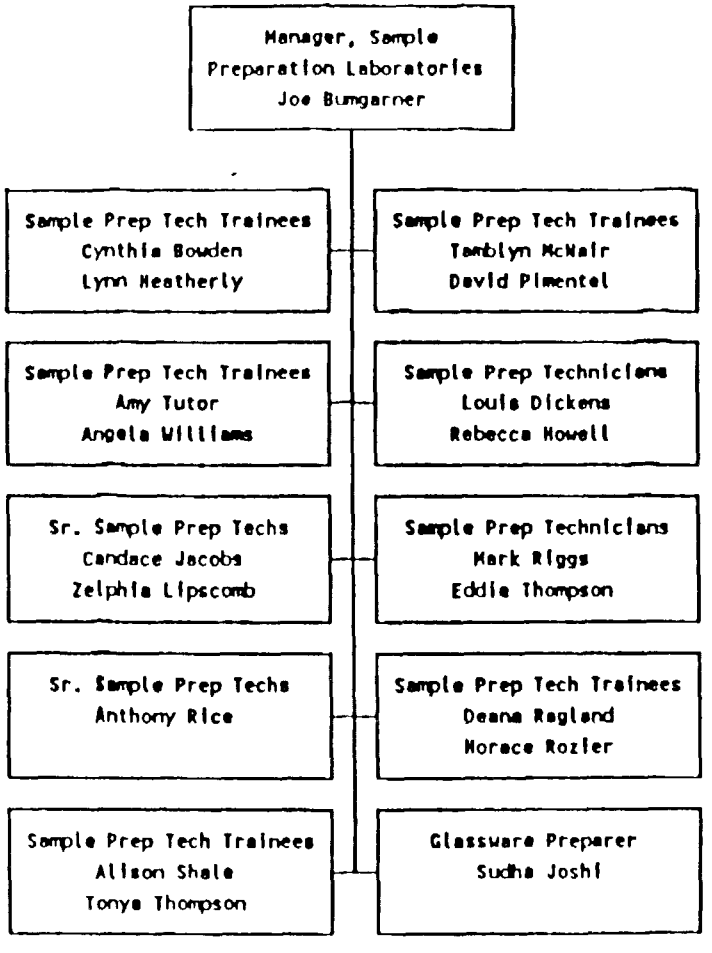


CompuChem Laboratories
Organics Laboratory
May 1, 1989




President & CEO
Kees Verkerk
Vice President,
Quality Assurance
Bob Meierer
Administrative Assistant
Angela Perrey
Supervisor Communications
William Morton
Manager QA
Bob Whitehead
QA Word Processor
Betty McIver
Communication Specialist
(vacant)
QA Coordinator
Shelby McCoy
QA Specialist
Peter Hill
Kathy Petersen
Sr. QA Specialist
Linda Fowler
Sr. Standard Tech.
Manhar Amin

CompuChem Laboratories, Inc.
Sample Preparation Laboratories
May 1, 1989



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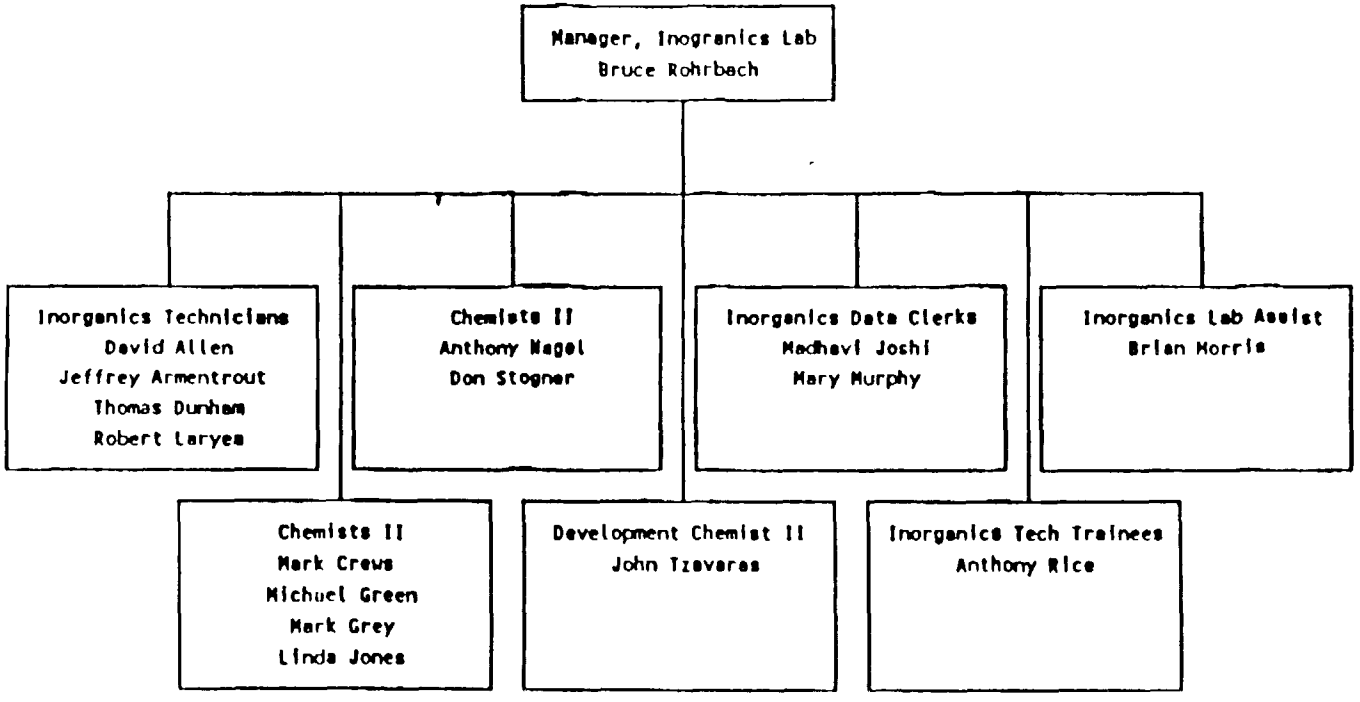
Inorganics Laboratory
May 1, 1989



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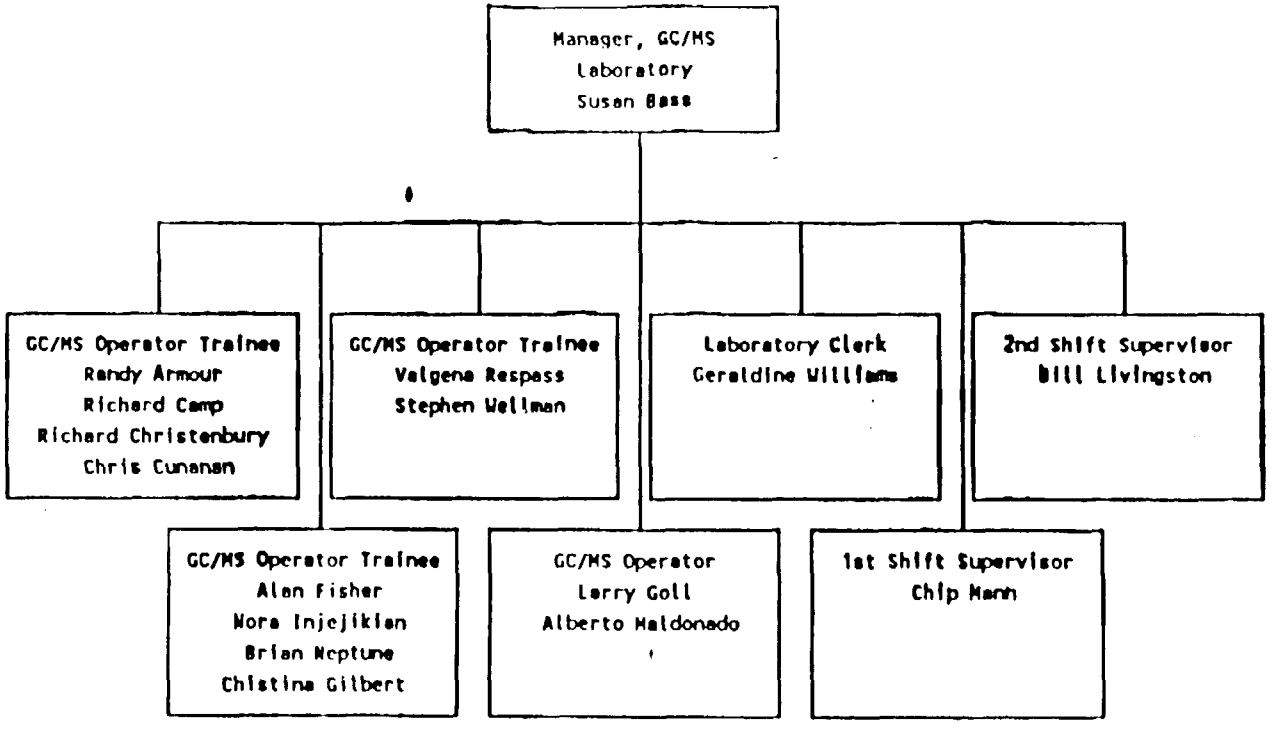
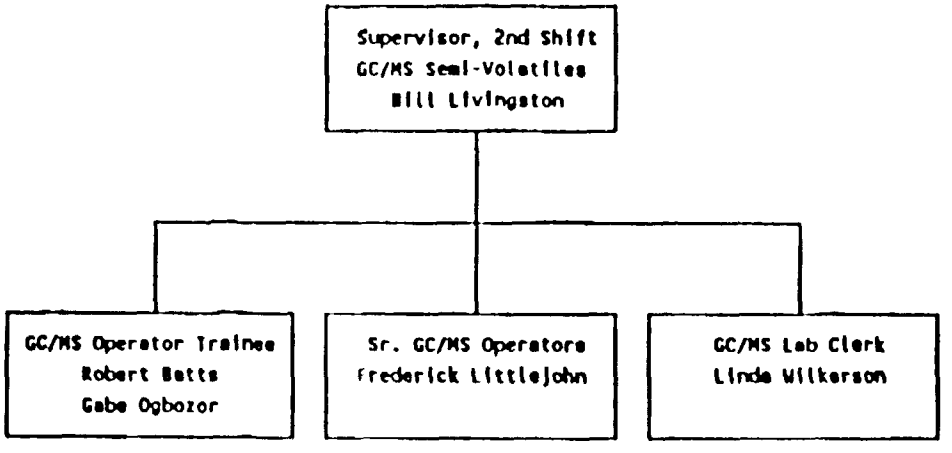
GC/MS Volatiles Laboratory
May 1, 1989



CompuChem Laboratories, Inc.
GC/MS Lab 2nd Shift
May 1, 1989



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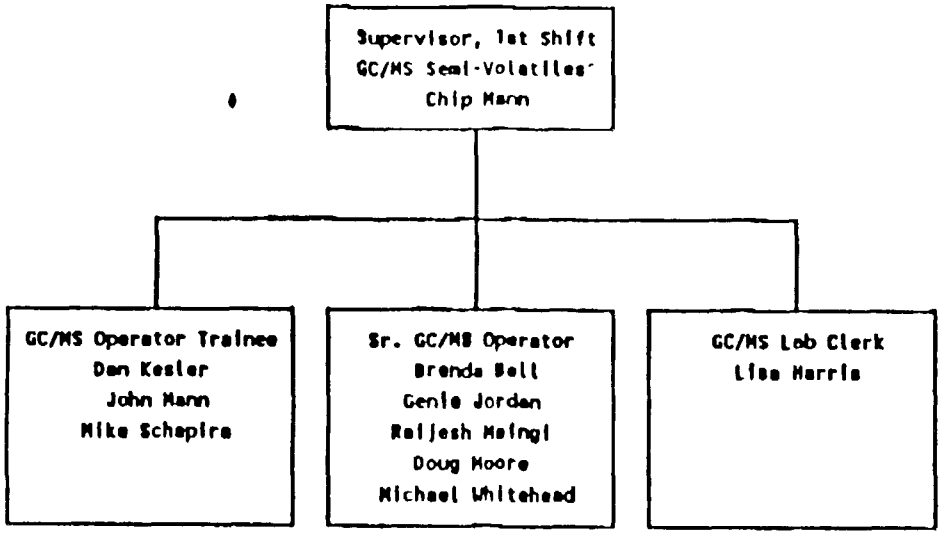
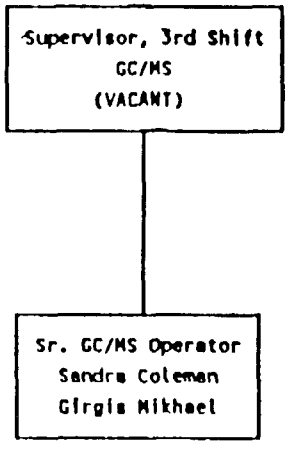
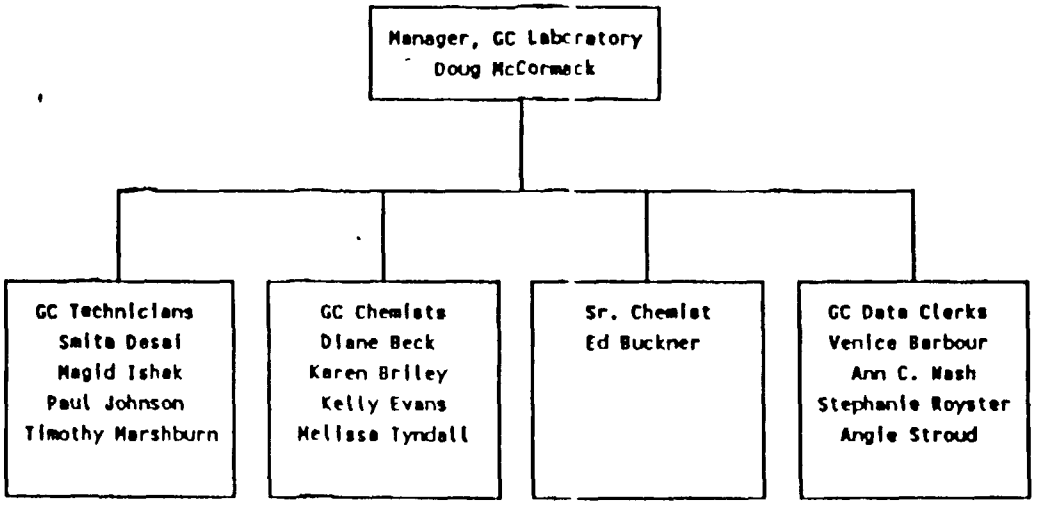
1st Shift GC/MS
May 1, 1989



CompuChem Laboratories, Inc.
GC/MS Lab 3rd Shift
May 1, 1989



CompuChem Laboratories, Inc.
GC Laboratory
May 1, 1989



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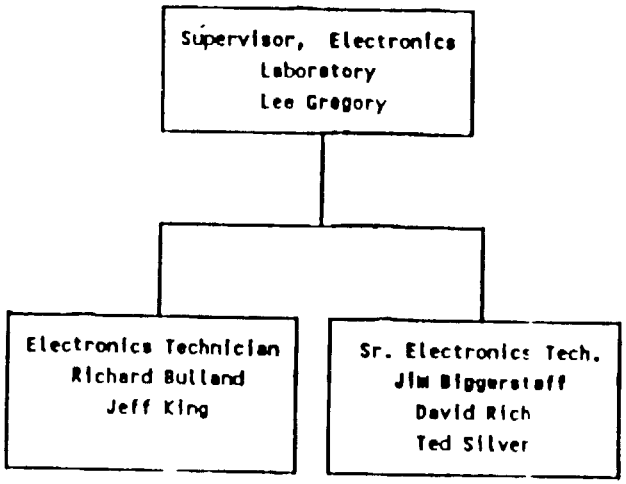

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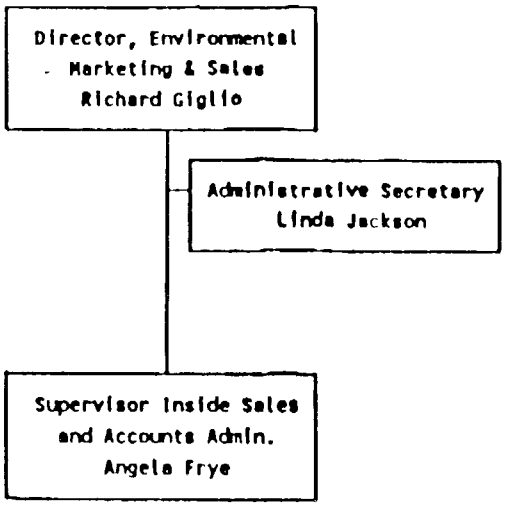

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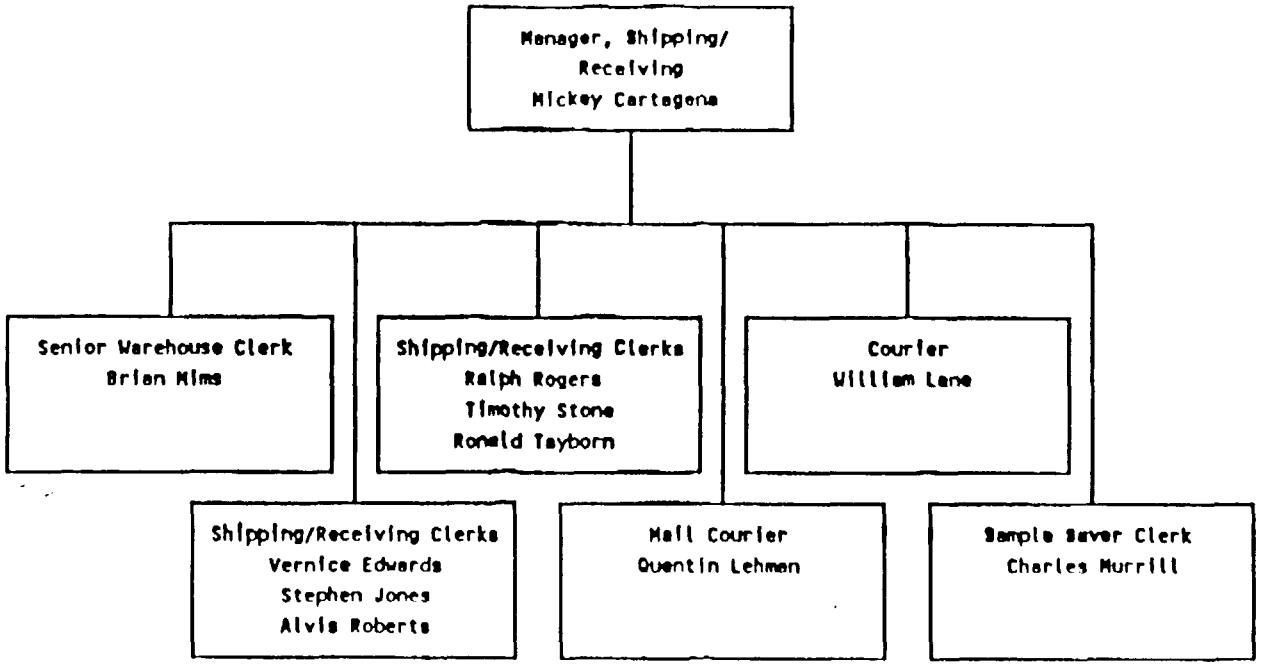
May 1, 1989



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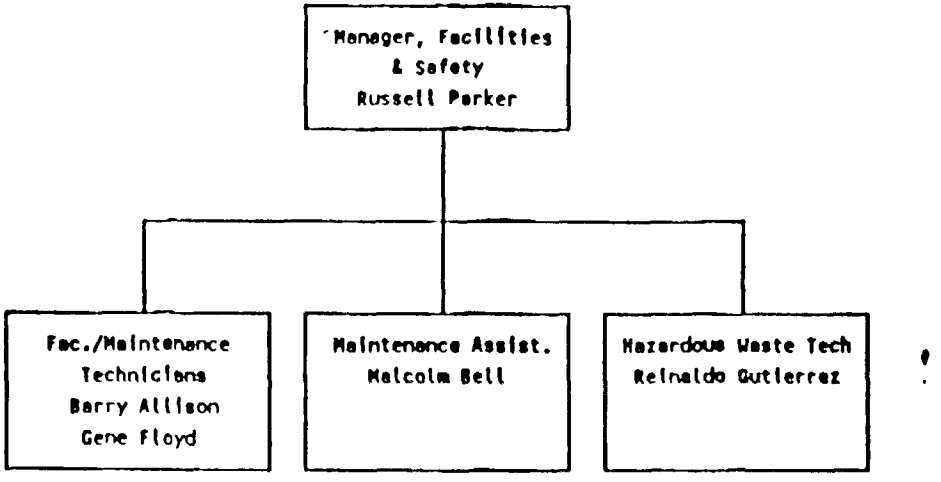
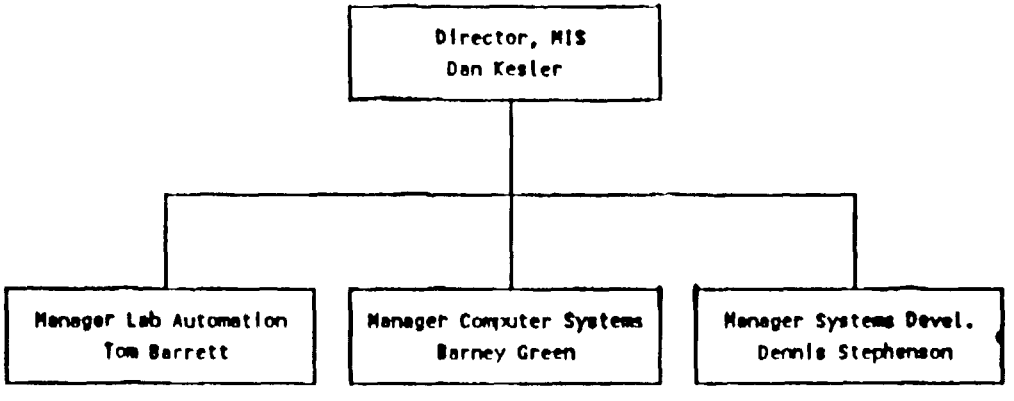


CompuChem Laboratories, Inc.
MIS
May 1, 1989



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Data Review and Final Technical Review
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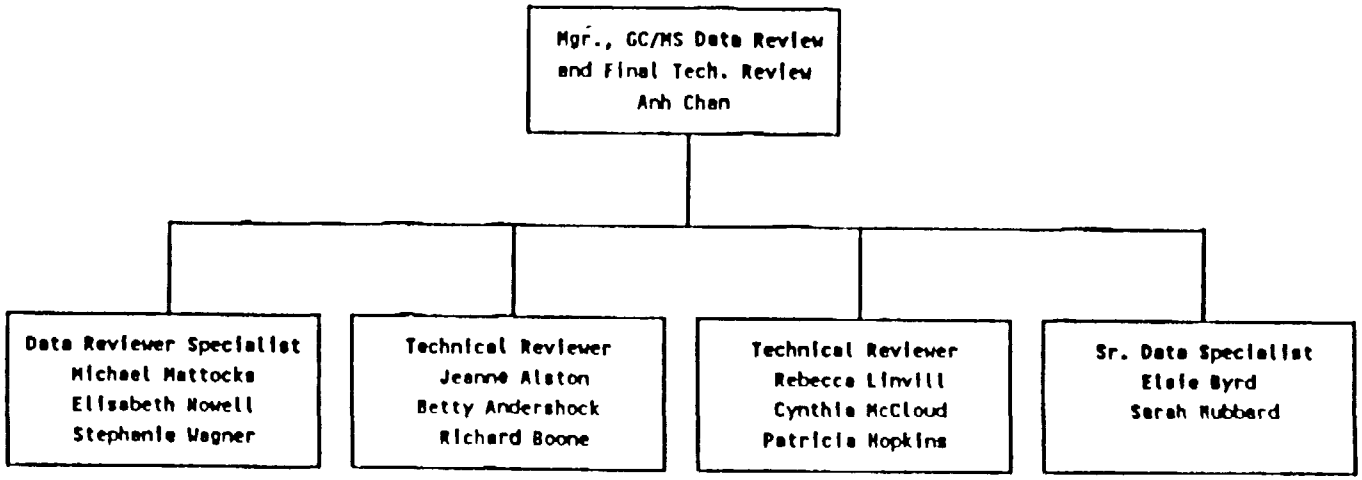


4.2 Assignment of Responsibilities

The main objectives of CompuChem's Quality Assurance Program are to assure that our laboratories generate data of known quality, that data quality meets or exceeds all QC/QA criteria, and that the records necessary to document laboratory performance are maintained. Additionally, the QA Department has the responsibility of providing feedback to management and to identify and implement policies to improve quality. The success or failure of the program is, to a great extent, dependent on the capabilities of those assigned the responsibility of carrying it out.

The following is a brief summary of the responsibilities and authorities assigned to each of the QA Department staff members:

Director of Quality Assurance

To be certain that the laboratory achieves all QA Program objectives, the Director of Quality Assurance monitors and directs the quality activities of QA Department and Lab personnel. The Director acts in strict adherence to the procedures and requirements stated in the Quality Assurance Plan.

The Director of QA reports directly to the corporation's CEO, is a member of the Executive Staff, and is organizationally and functionally independent of all personnel directly involved in the operation of the technical program. The Director has the authority to terminate non-conforming work at any time.

Additional responsibilities and duties include:

Preparing and overseeing the preparation of the QA Plan;

Monitoring the QA Program as documented in the QA Plan and ensuring that all elements are carried out as written;

Developing and implementing new QA programs, including statistical procedures, additional quality control measures, validation of new methods, etc.;

Conducting scheduled or unannounced audits and inspections, reporting findings to management, and when needed, ensuring that corrective action is taken;

Maintaining current documentation of all measurement procedures routinely used in the laboratories, including those used by subcontractors;

Periodically informing management of the status of the QA Program;

Seeking out and evaluating new ideas and current developments in the field of QA and recommending means for their application where advisable;

Implementing or modifying analytical methods to conform with recognized standards and/or fundamentals of Good Laboratory Practices, including alteration of analysis/procedure codes used by the Computerized Laboratory Management System (CLMS);

Final authority to terminate or alter any incorrect or improper analytical or measurement procedure in order to conform to requirements of the QA Plan;

Training, directing and qualifying personnel in specified laboratory QC and analytical procedures, or designating qualified individuals to do so;

Review and advise lab management on requirements and applicability of specific Quality Assurance Project Plans (QAPPs), new statements-of-work (SOWs), RFQs, RFPS, IFBs, and other contract-related issues.

Reviewing corrective action reports for out-of-control events and verifying that remedial action has been taken to restore control;

Assuring that subcontractor laboratories are complying with the QA Program;

Serving as point-of-contact for exchange of QA/QC information and approving, along with the Laboratory Director, release of QA/QC information;

Direct the activities of the QA Department technical staff and Communications Specialist.

Manager of Quality Assurance

The Manager of Quality Assurance reports to the Director of Quality Assurance and functions independently from the laboratory technical program. The Manager is primarily responsible for overseeing and directing the activities of the Quality Assurance staff, consisting of Sr. QA Specialists, QA Specialists, Sr. Standards Chemists, and clerical staff. The Manager of QA, the QA Specialists and the Sr. QA Specialists have authority to approve data. Other than these individiuals, only the Final Tehcnical Reivewer has the authority for final data approval.

Additional responsibilities and duties include:

Provide QA reports to management, as described in Section 10.0;

Oversee the laboratory's participation in external QA/QC programs;

Coordinate external (on-site) or internal QA/QC audits or inspections;

Review and approve all laboratory-generated data qualifying notices;

Write Quality Assurance Notices, used to document exceptions to QC acceptance criteria or other matters affecting data useability or interpretation, for inclusion in data packages;

Provide training to QA and laboratory staff;

Provide assistance on special projects as required by the QA Director;

Oversee all subcontractor QA Programs, including administration of subcontractor proficiency studies and on-site audits;

Assume the responsibilities of the QA Director, if necessary.

Quality Assurance Staff

The Quality Assurance Staff is responsible for carrying out quality activities as directed by the QA Manager and QA Director. All members of the Quality Assurance staff function independently from the laboratory technical programs.

Additional responsibilities and duties include:

Create and revise SOPs, including integration into proper document control format, maintaining a chronology and distributing SOPs to laboratory stations;

Ensure that the laboratories meet all requirements as documented in the QA Plan, as well as the specific QA and lab SOP manuals;

Audit and spot-check work in process for quality and completeness;

Provide deviation/exception reports to laboratory managers and the QA Director regarding out-of-control analyses and provide recommendations for corrective action;

Oversee corrective action as required;

Generate, analyze and document QC/QA data (much of the QC data is generated by the laboratory staff in the normal course of producing analytical data, or by using the CLMS during data acquisition or data entry);

Based upon laboratory performance statistics and/or statement-of-work requirements, establish and update control limits using QC data from routine sample analyses;

Provide information and documentation for internal/external audits or inspections;

Function as a liaison between the QA Manager/Director and personnel within the laboratories;

Communicate QA Program objectives and requirements to clients and external auditors;

Reinforce GLP within the laboratory;

Communicate any quality concerns to the QA Manager/Director;

Communicate any safety concerns to the Manager of Facilities and Safety;

Review and approve Performance Evaluation (PE) and Proficiency Testing (PT) sample data;

Review PE and PT scores, coordinating lab personnel review of unacceptable scores and associated data, and assembling findings into unified document for response to certifying agency;

Initiate and document corrective action (if necessary) related to unacceptable scores and associated data, and assembling findings into unified document for response to certifying agency;

Initiate and document corrective action (if necessary) related to unacceptable PE/PT scores or audit deficiency reports;

Introduce internal "blind" PE samples into the CLMS (or mailing to subcontractor labs) and report performance to management (this includes both initial and continuing certification of performance, and is required of all subcontractor laboratories);

Document approval and traceability of all standards to NBS, USEPA or other certified source;

Document calibration traceability of all thermometers, balances and Class-S weights used in daily calibrations;

Audit the Environmental Site Profile (remote reporting) database, summarizing findings in reports to management;

Conduct routine Performance Audits and System Audits, summarizing findings in report to management;

Audit computer output tabulating all keycard accesses to restricted areas, summarizing findings in report to management.

Laboratory Personnel and Management

A variety of Quality Control functions and duties directly or indirectly affecting data quality are performed by laboratory personnel and management.

Key responsibilities of this nature include:

Perform scheduled, routine preventative maintenance or instruments or oversee work done under service contracts;

Assure compliance with methods and SOPs and as written as directed by the Quality Assurance Director;

Verify that all instruments meet calibration and tuning requirements;

Respond to corrective action requirements;

Follow GLPs and recommendations of the QA Department for improving quality and safety;

Perform and document action steps based on established QC acceptance criteria;

Provide adequate and documented training of personnel.

4.3 Communications

The Quality Assurance Department communicates to other areas of the laboratory and to Management via several different types of reports, the key QA reports as described in Section 10.0 . The Director of QA and the QA staff also distribute inter-office memoranda to appropriate laboratory management detailing the results of internal and external audits, blind interlaboratory proficiency studies, blind internal proficiency studies, and deficiency reports/corrective action needs. Good Laboratory Practices and successful performance on various studies and audits are also reinforced through these memoranda.

Section 10.0 provides additional details regarding the monthly and quarterly reports distributed to all laboratory managers and appropriate senior and executive staff.

4.4 Document Control

Document control procedures, as described in section 1.4.1 of the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I (EPA-600/9-76-005), are used in the production of the QA Plan and other documents vital to the operation of the laboratory. This document control system, described in the QA SOP Manual, includes official distribution lists, a historical file of all updated standard operating procedures, and appropriate signature levels for the ensurance of correct methods, procedures and techniques.

4.5 Personnel Qualifications

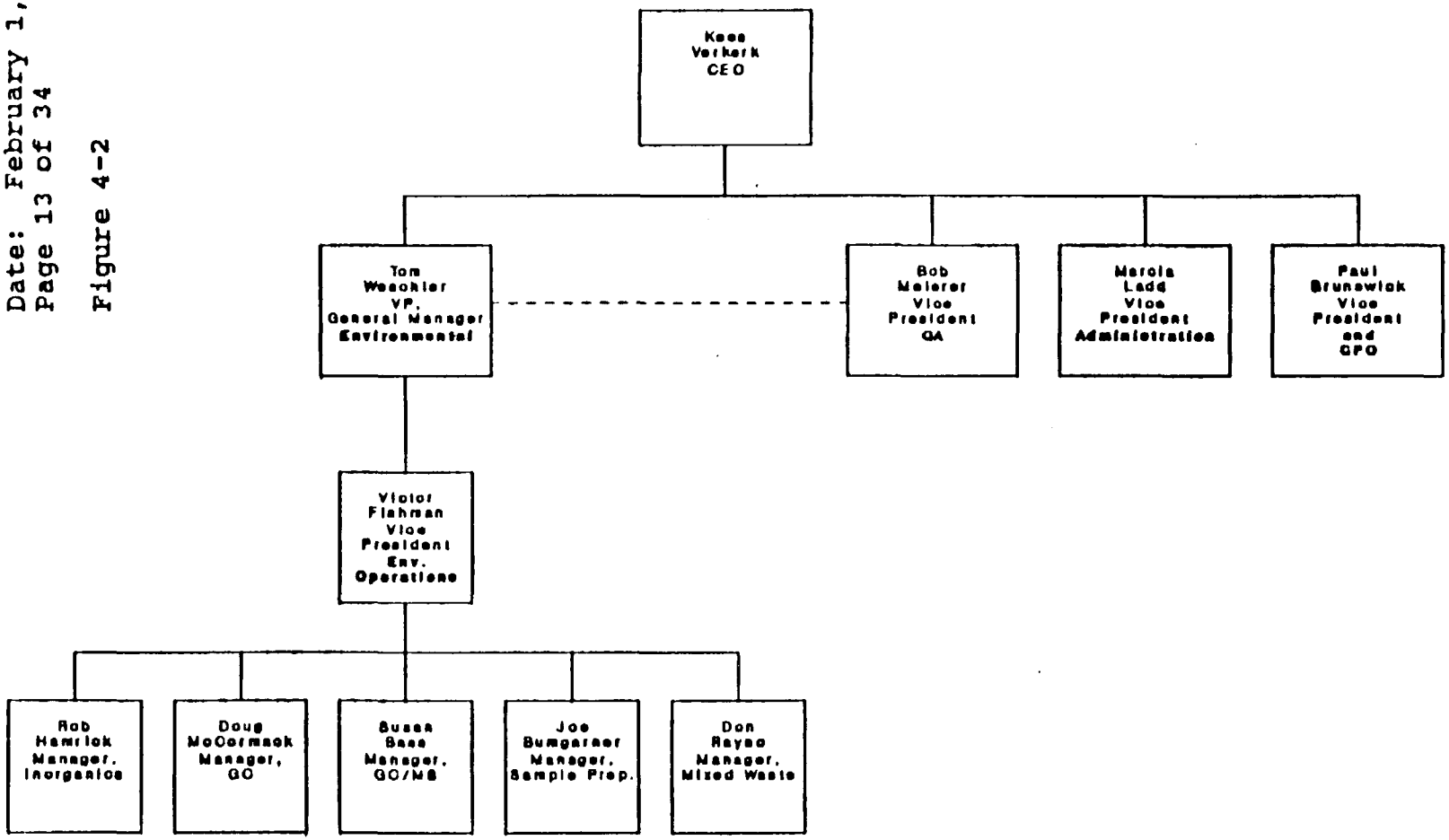
CompuChem, located in Research Triangle Park, NC, and within minutes of three major university campuses, is ideally positioned for recruiting both degreed scientists and experienced professionals. Many applied science graduates join the organization as entry-level technicians, and progress through extensive training into senior chemist, data review/validation, and QA positions. In most technician positions and instrument operator positions, the training period lasts from six months to one year, depending on the level of experience required and complexities of the position/instrumentation.

Job descriptions, including required educational, training and experience qualifications, are incorporated into individual personnel records and are available for review in the Human Resources Department. These records also serve to document additional cross-training and participation in continuing education programs.

QA Plan users are referred to Section 17.0 for resumes of key personnel. Also included in this are the USEPA's requirements for qualifications of technical personnel involved in the analysis of EPA samples. CompuChem's technical personnel meet or exceed these requirements in all cases. Suitable staff are nominated in the QA SOP to deputize for senior technical staff and QA management in their absence.

Figure 4-2

Organization Chart
Role of QA in Env. Lab Operations

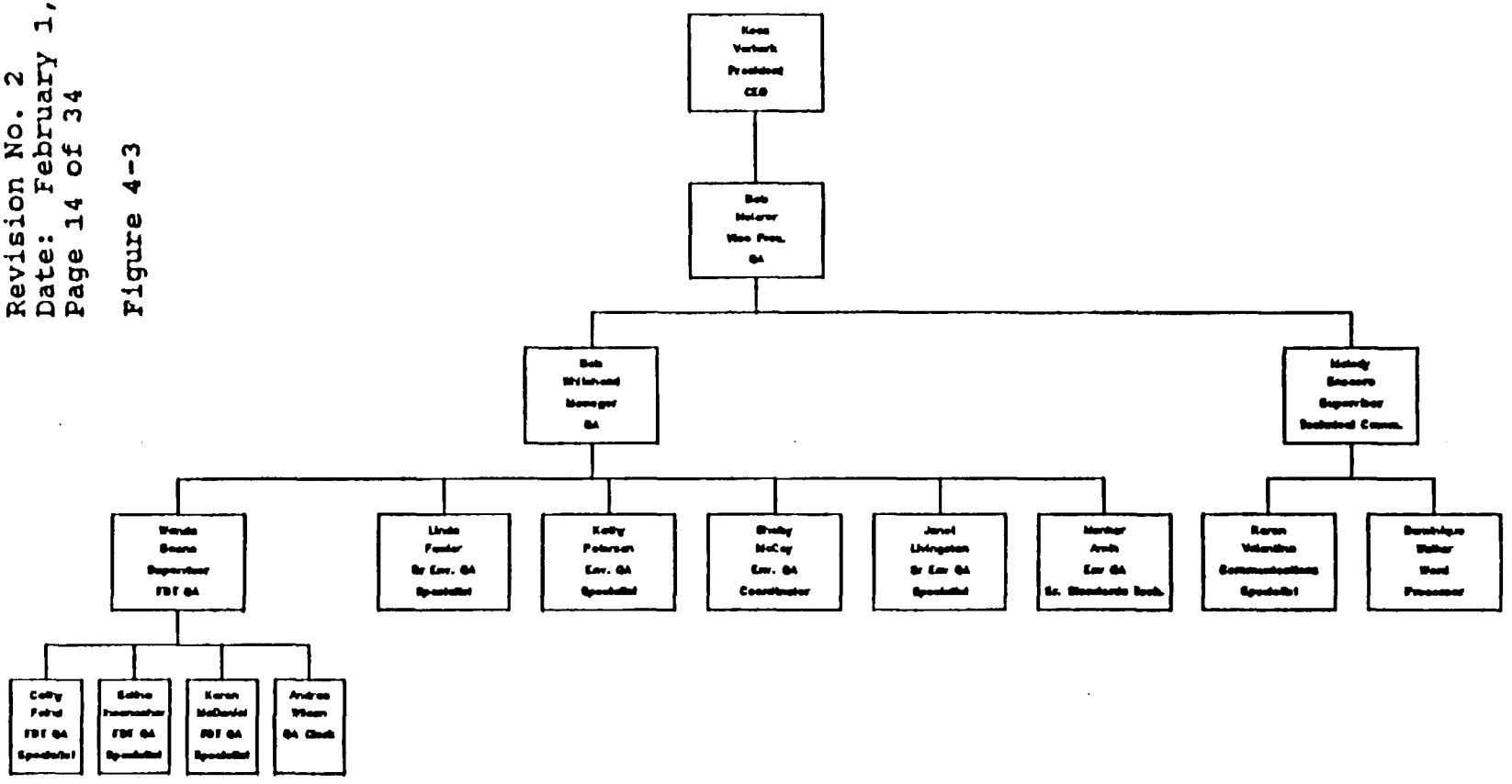


January 1991

Figure 4-3

Corporate Structure

Quality Assurance

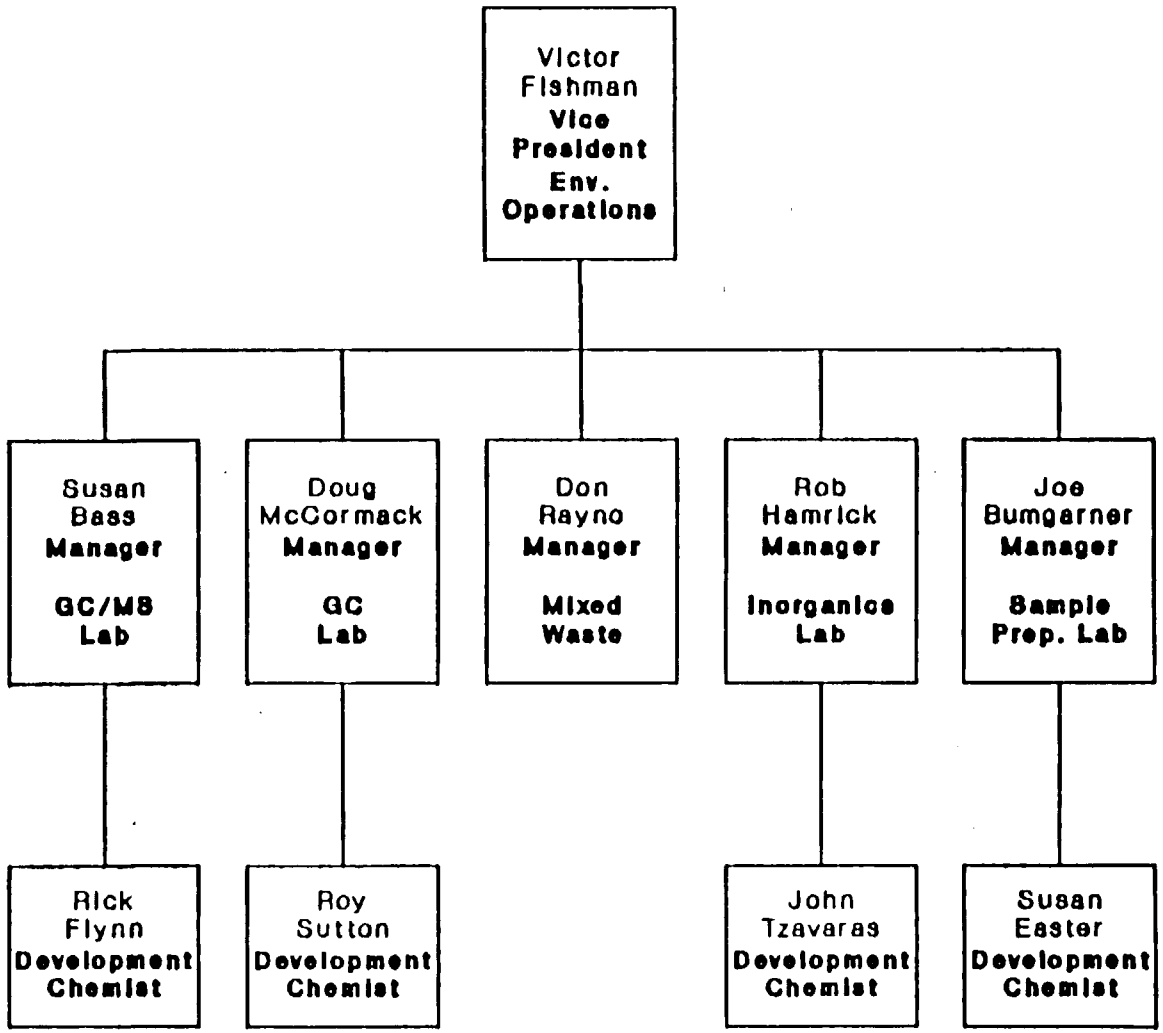


January 1991

Figure 4-4

Organization Chart

Lab Mngmnt & Assoc. Development Chemist



January 1991

Figure 4-5

Organization Chart

Lab Instrumentation

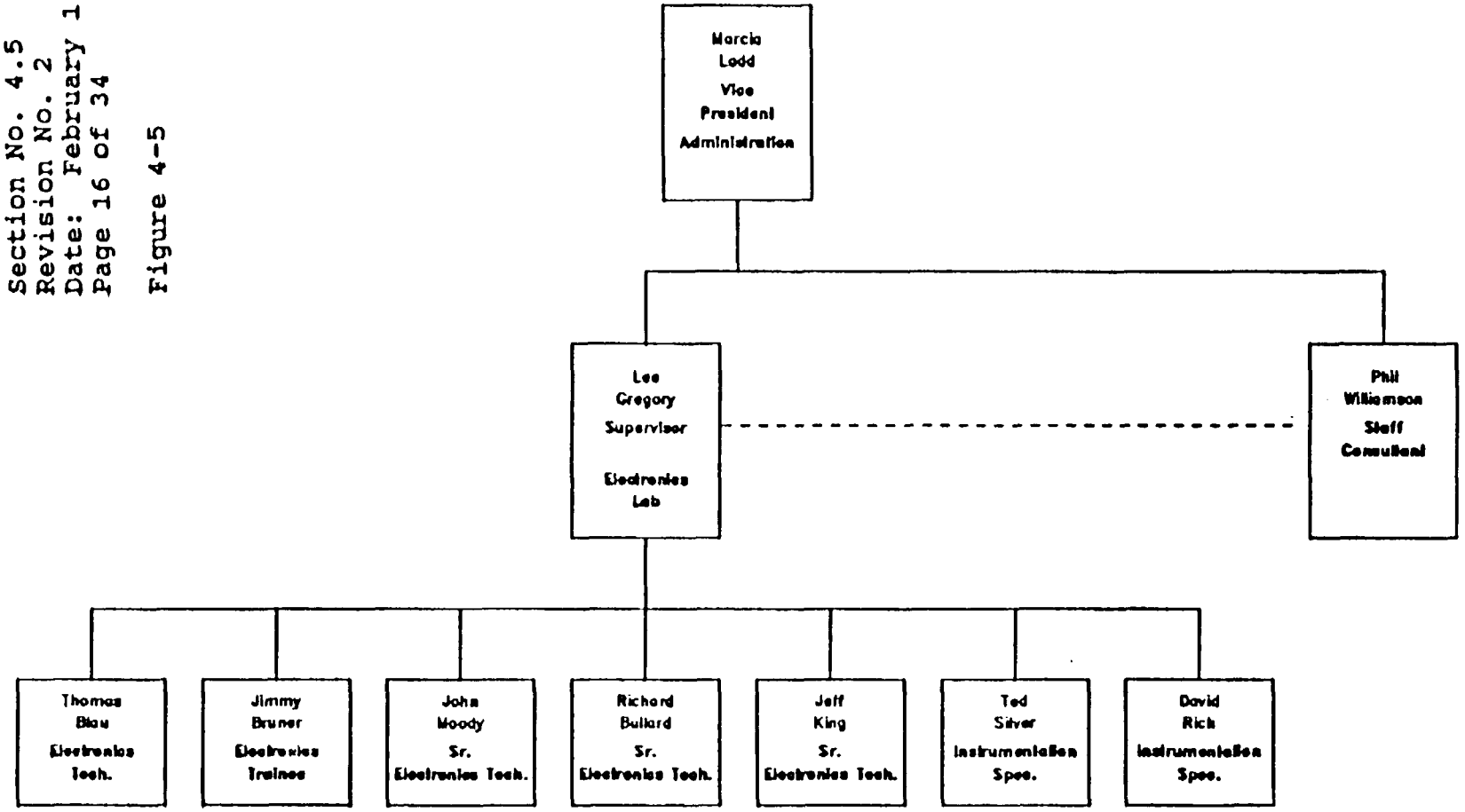


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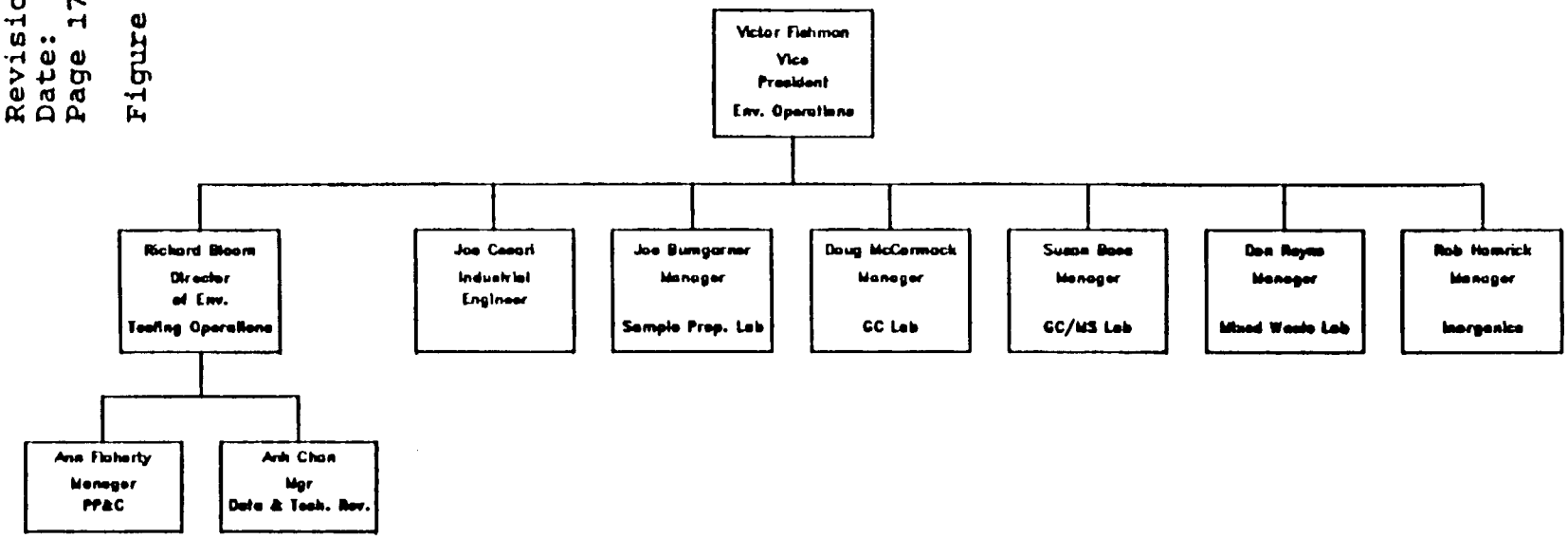
Figure 4-6

Organization Chart

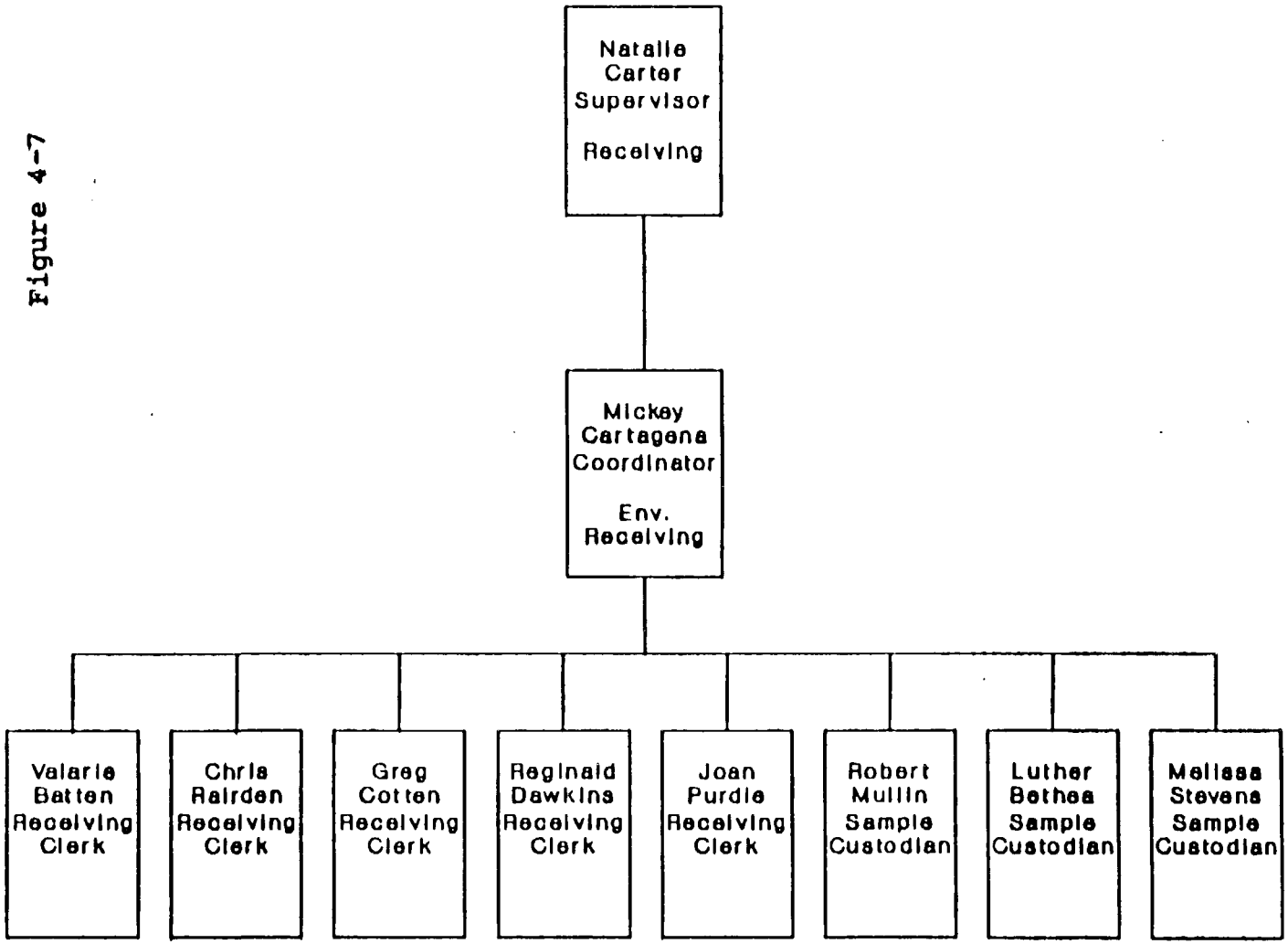
Environmental Operations



January 1991

Figure 4-7

Organization Chart
Receiving



January 1991

Figure 4-8

Organization Chart

Report Deliverables

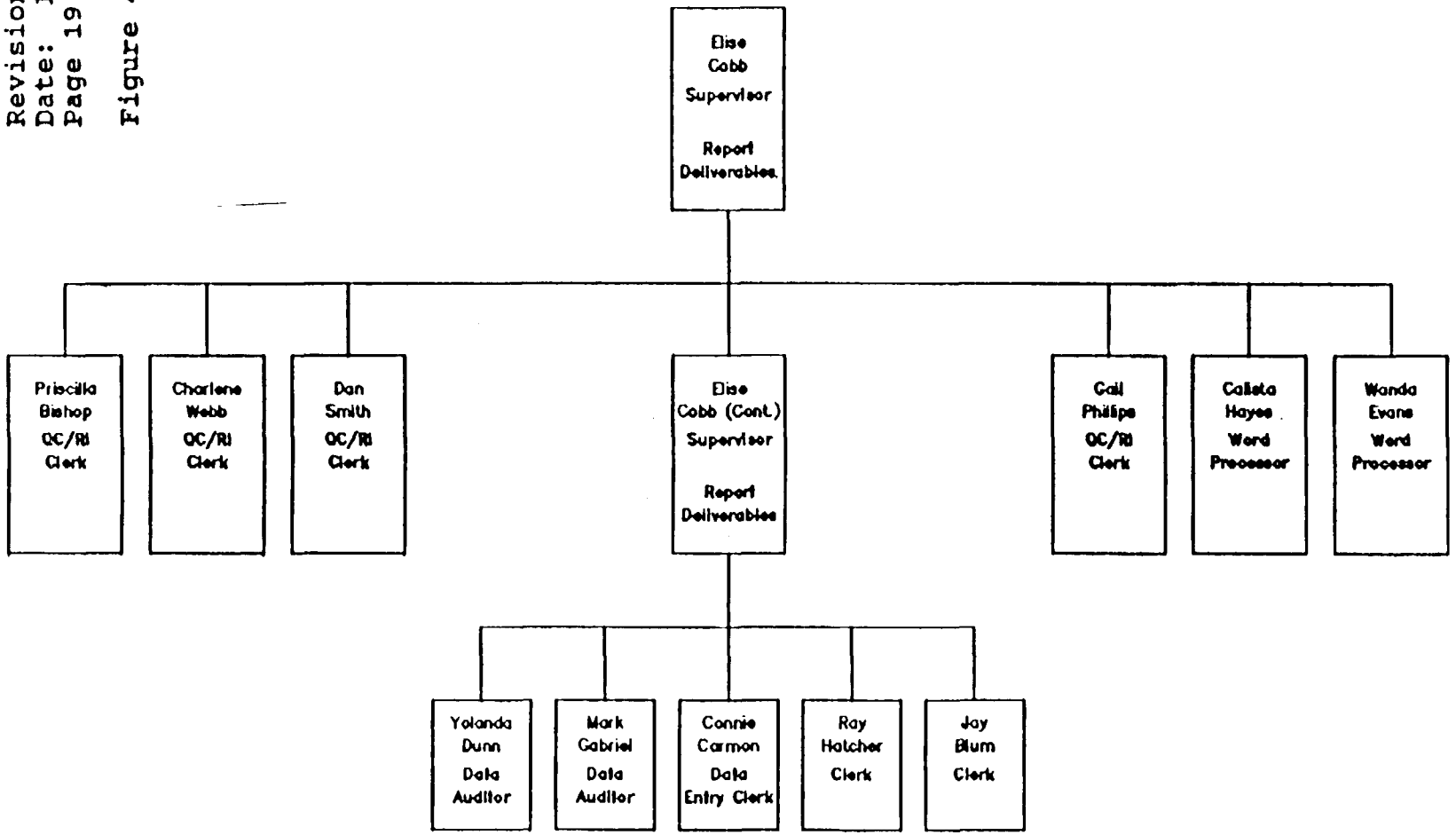


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Figure 4-9

Organization Chart

GC/MS Data Review

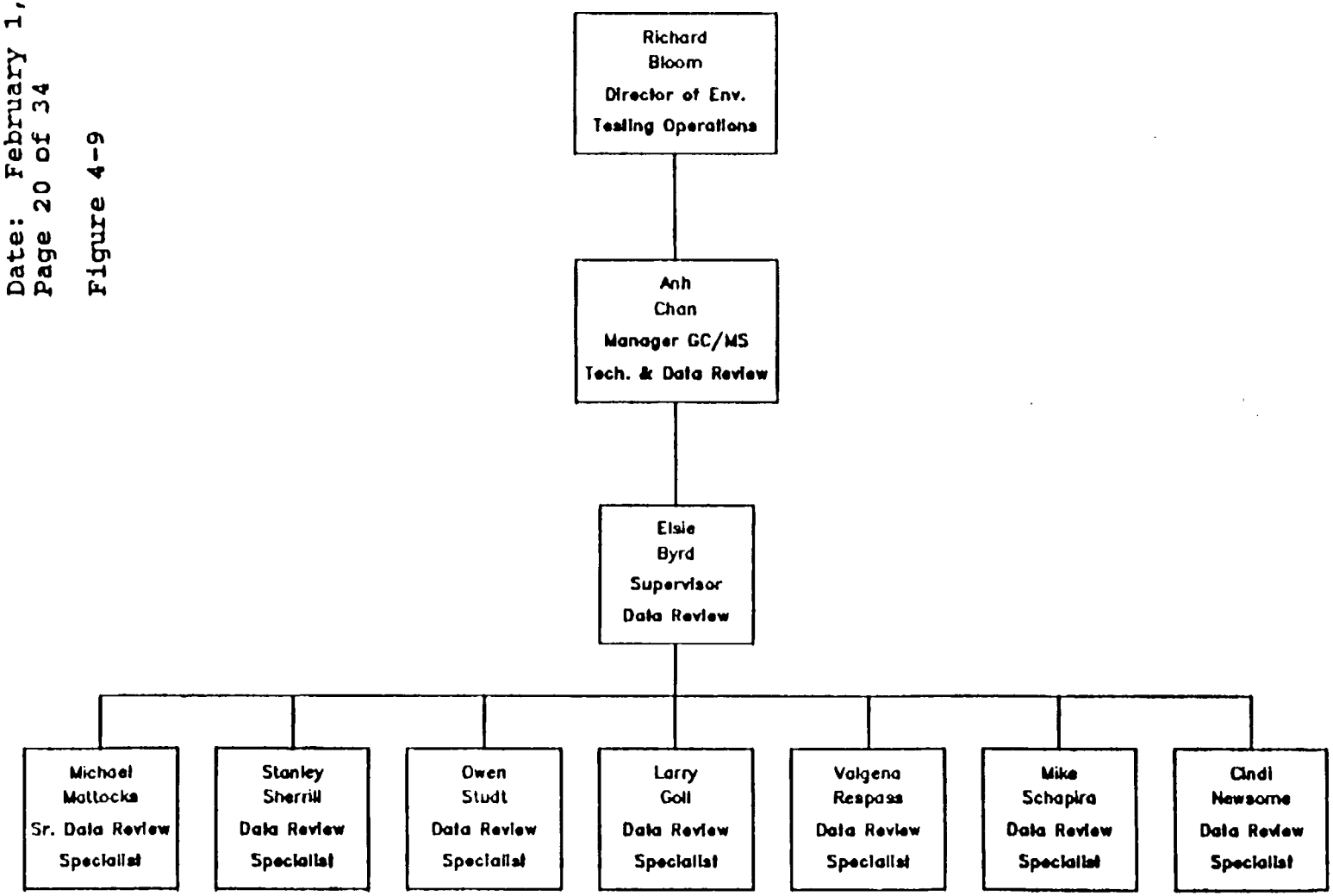

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Figure 4-10

Organization Chart

Final Technical Review

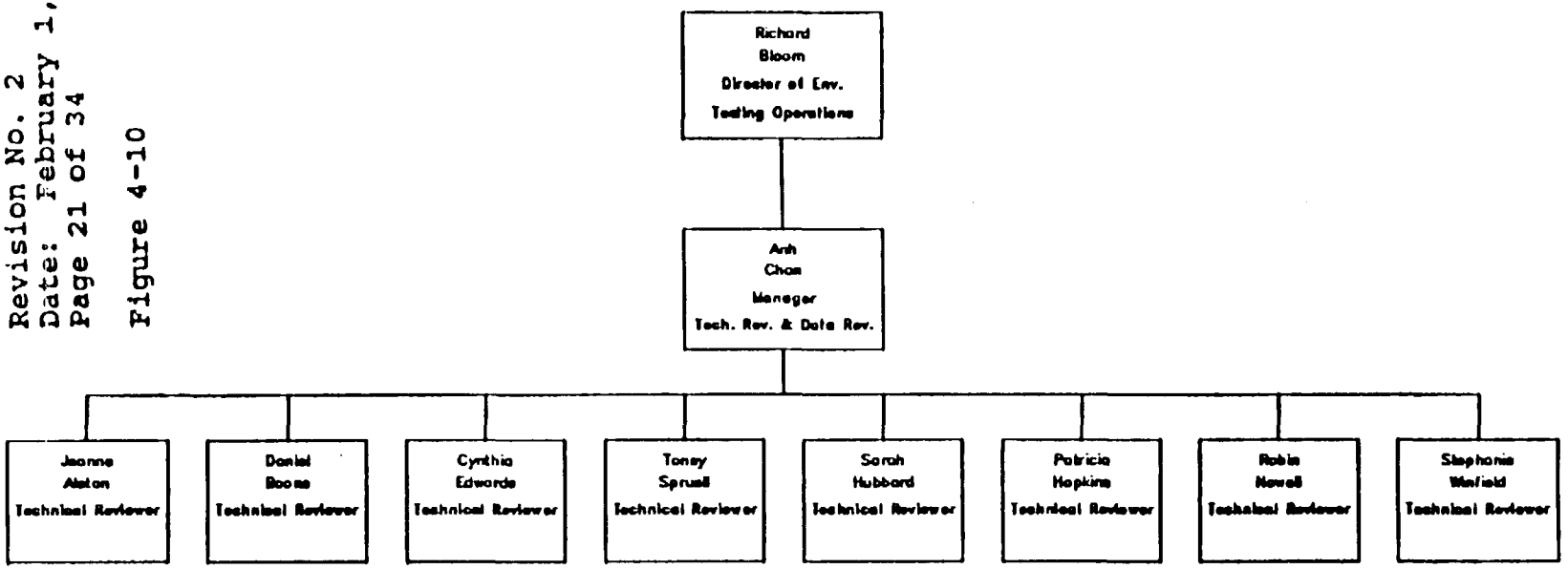

Figure 4-11

Organization Chart
Sample Preparation Lab/1st Shift

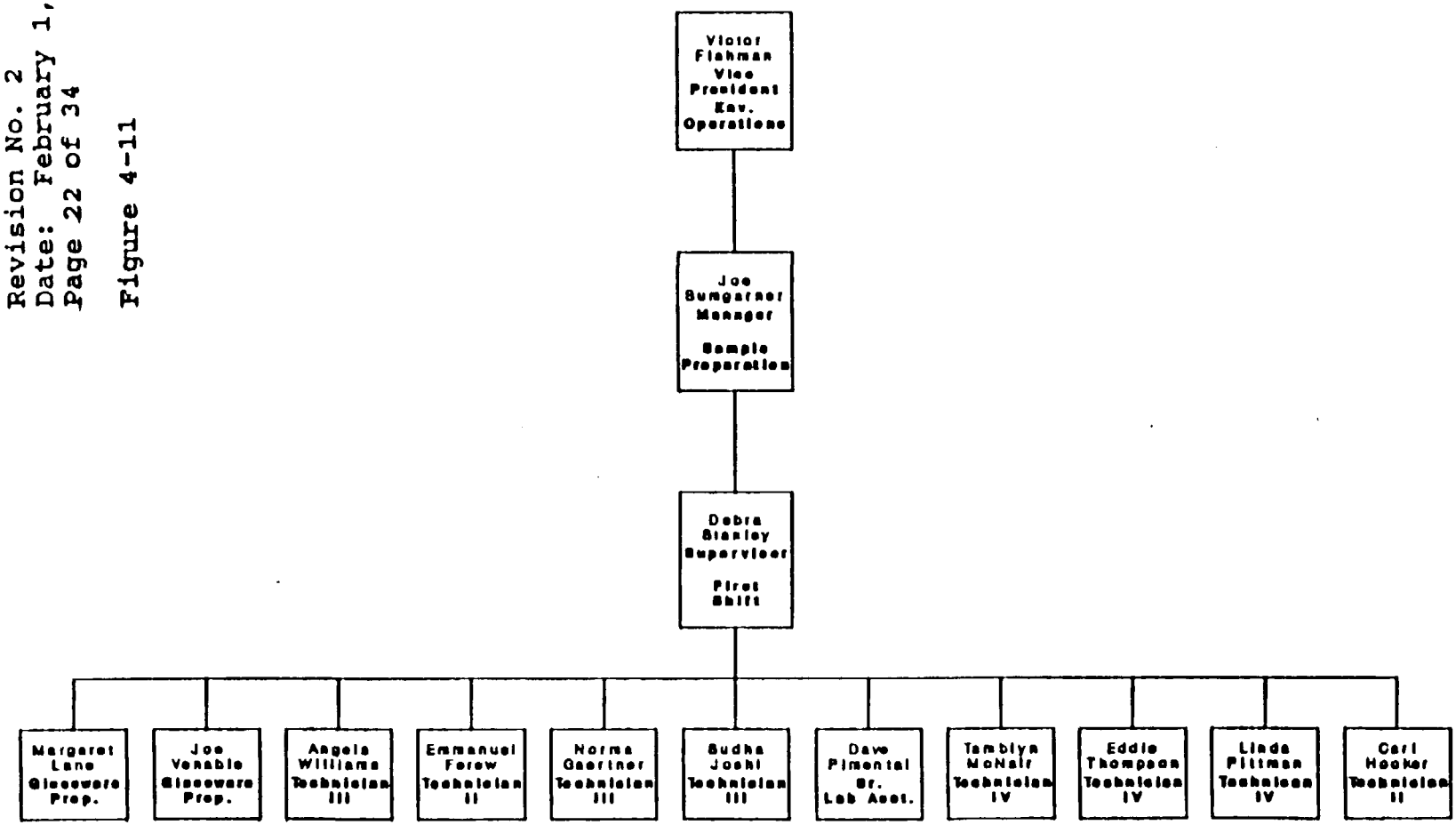

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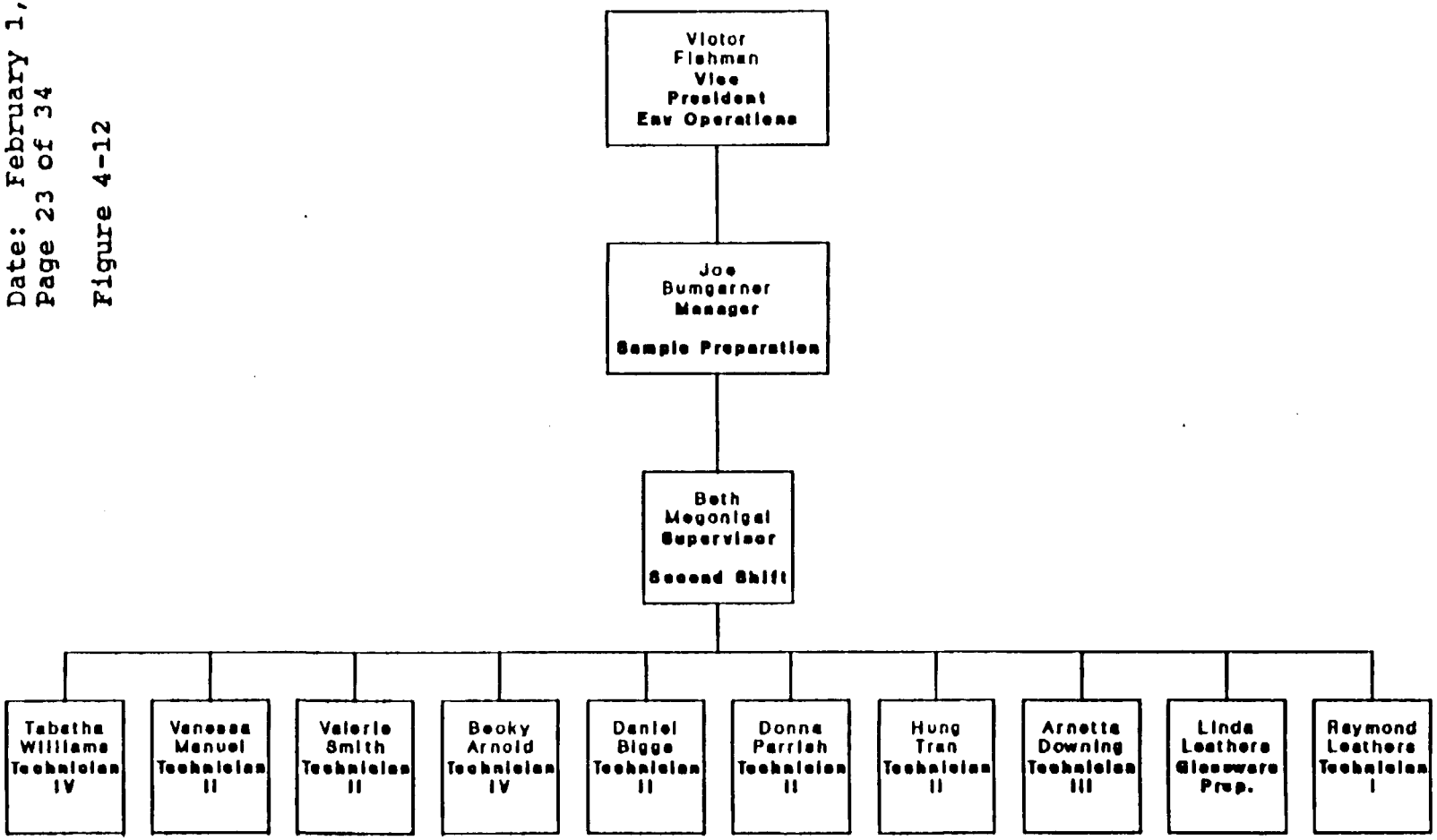
Figure 4-12

Organization Chart
Sample Preparation Lab/2nd Shift



January 1991

Figure

Joe
Bumgarner
Manager, Sample Prep.
& Org. Char. Lab
Patrick
Conarton
Chemist II
George
Griffith
Chemist I

Figure 4-14

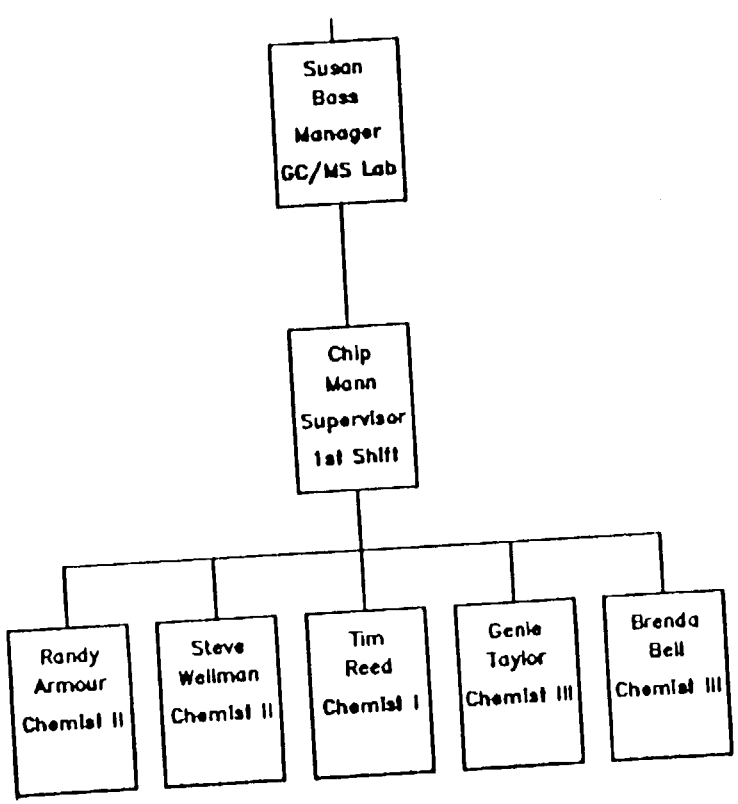
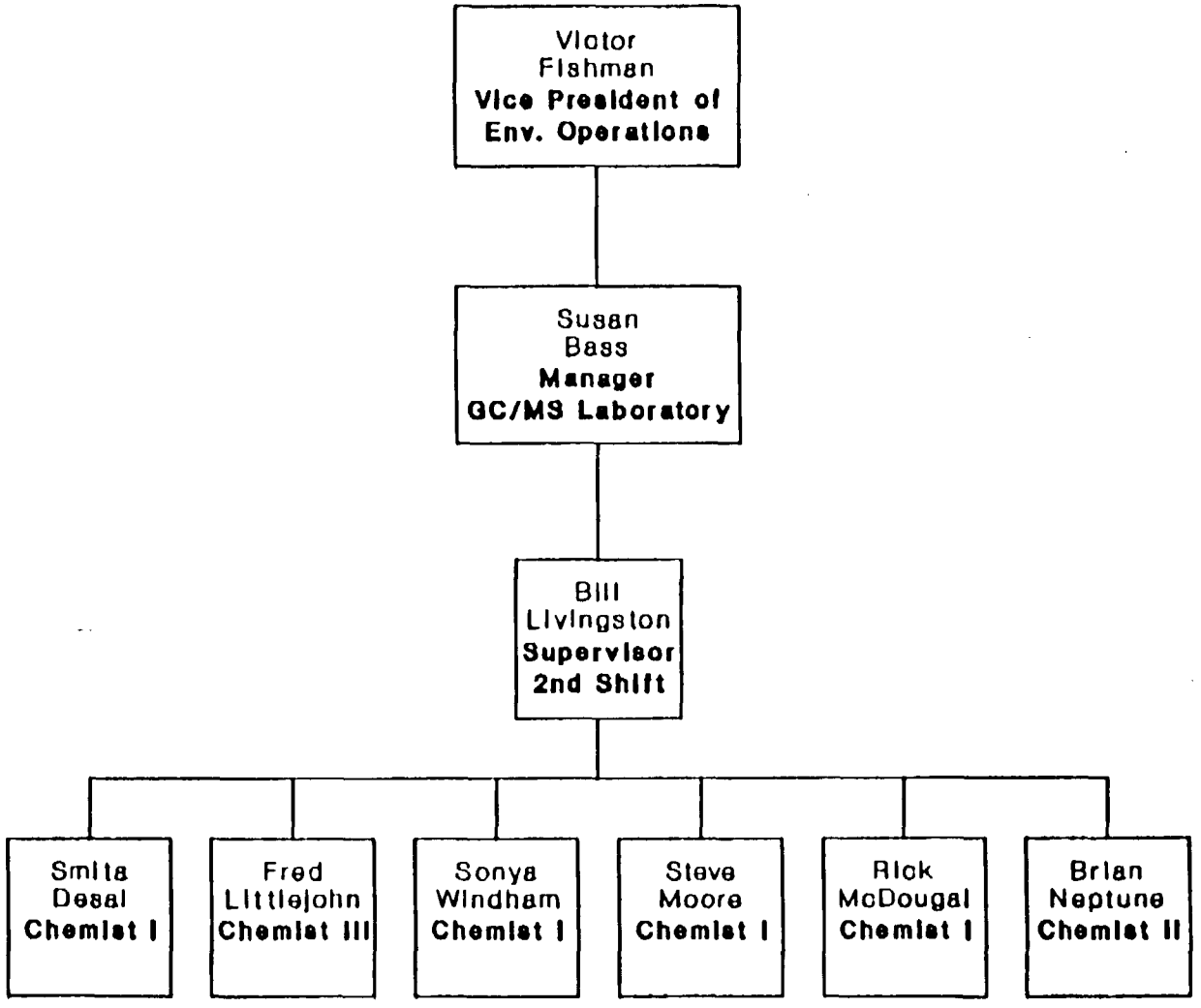

Susan
Bass
Manager
GC/MS Lab
Chip
Mann
Supervisor
1st Shift
Randy
Armour
Chemist II
Steve
Wellman
Chemist II
Tim
Reed
Chemist I
Genie
Taylor
Chemist III
Brenda
Bell
Chemist III

Figure 4-16

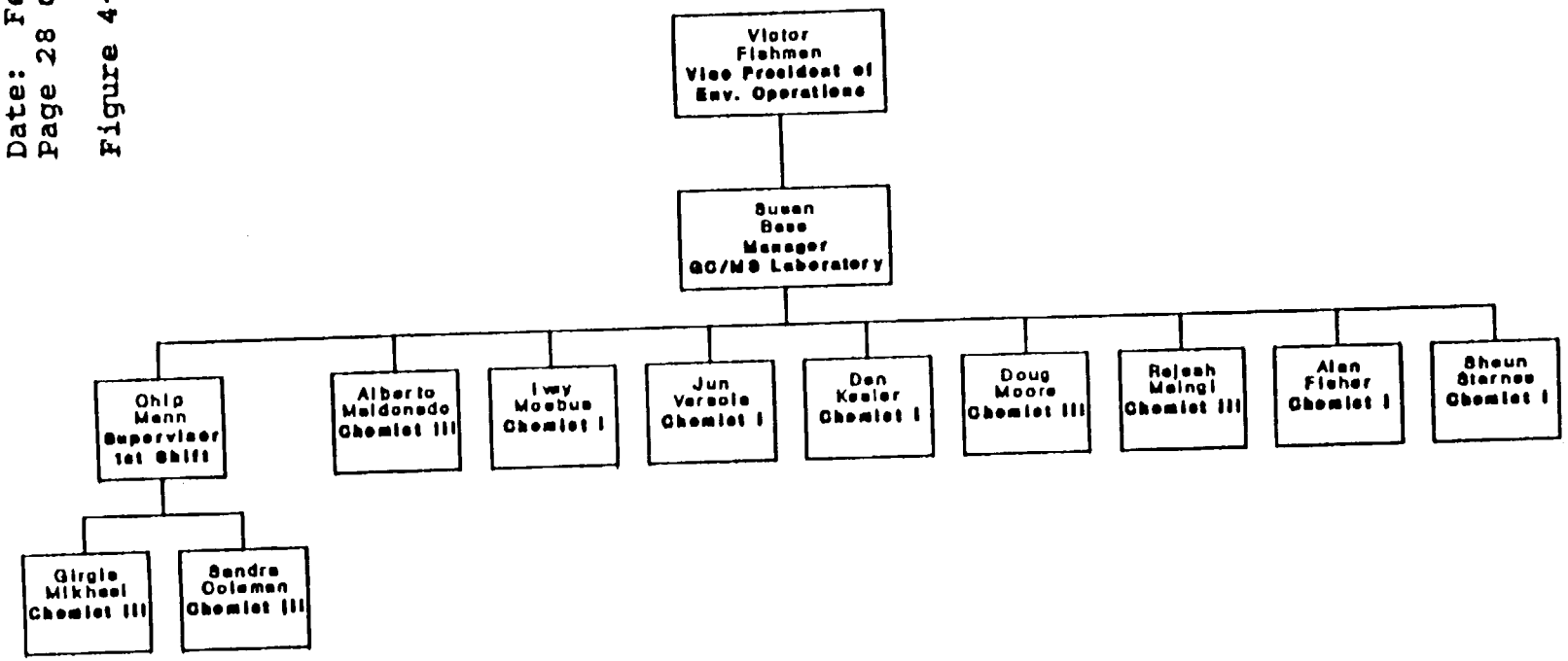
Organization Chart
GC/MS Laboratory/2nd Shift



January 1991

Organization Chart
GC/MS Laboratory/3rd Shift, Weekends

Figure 4-17



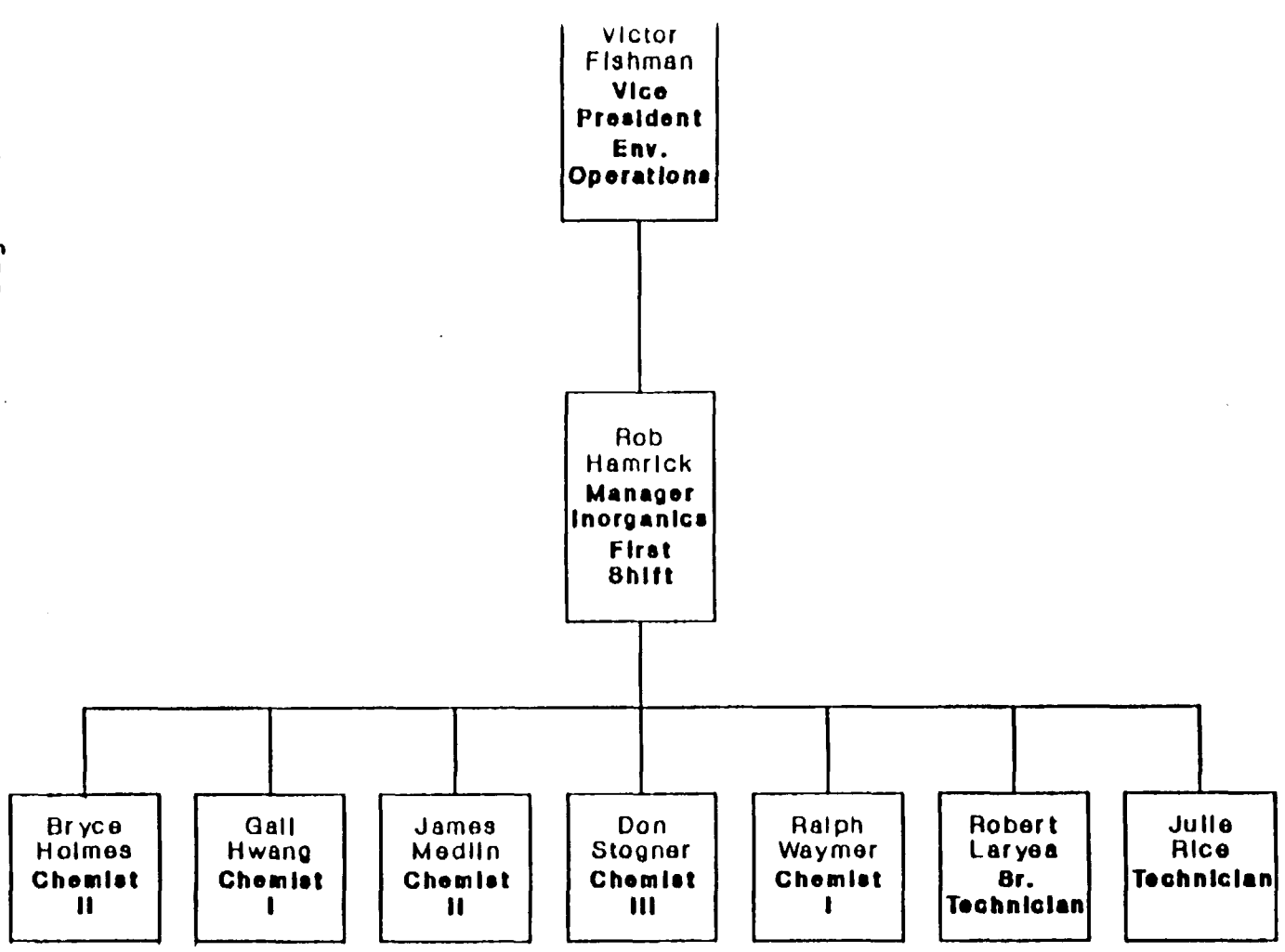
January 1991

Figure 4-18

Organization Chart
Mixed Waste Lab

January 1991

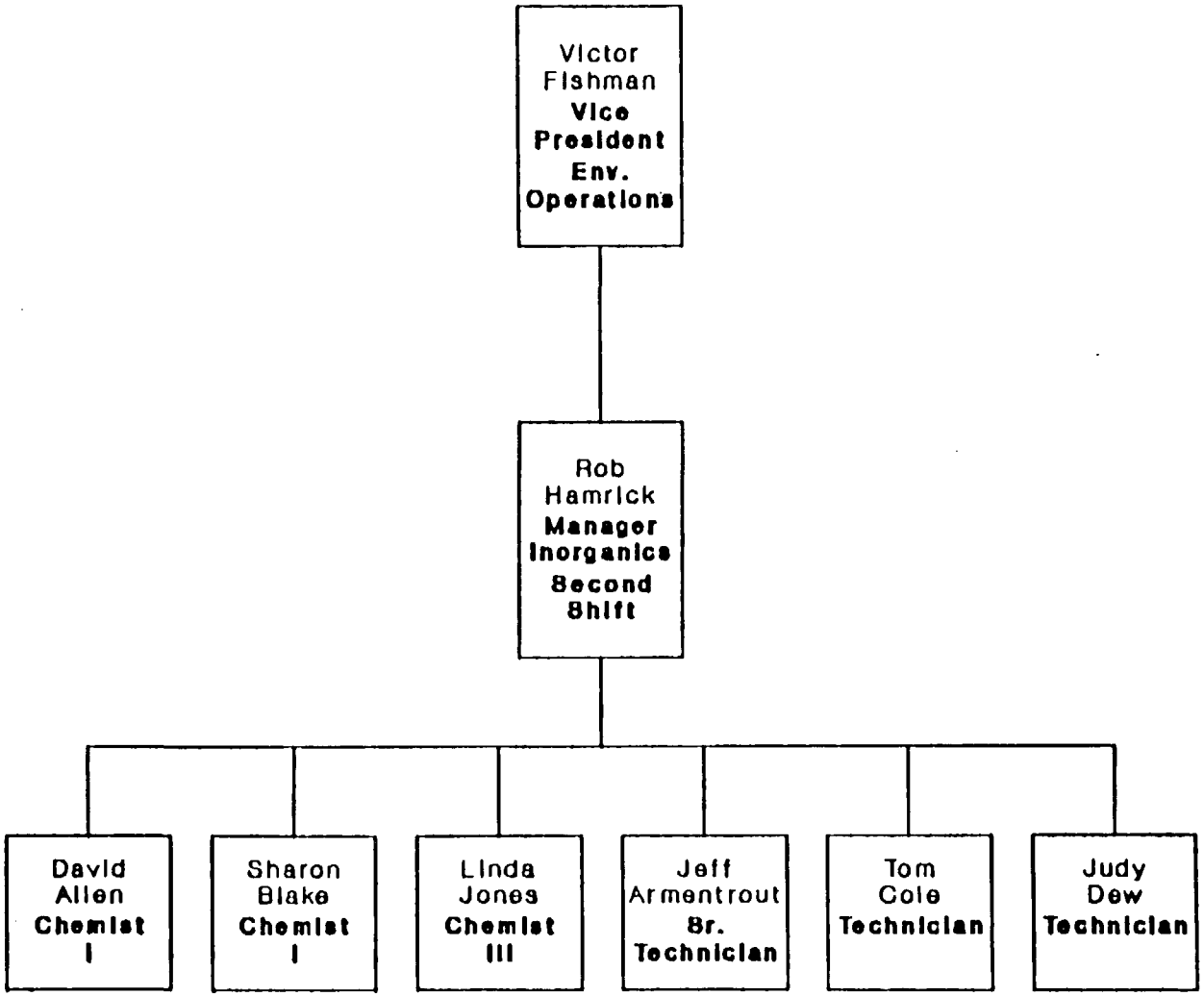
Figure 4-19



January 1991

Figure 4-20

Organization Chart
Inorganics Laboratory



January 1991

4.5 QA Program Assessment

The Director of QA and the department staff conduct periodic assessments of the total QA program. Based on these assessments, a written status report of QA activities and progress is forwarded to management. The following items are addressed in these reports, most being addressed in the Monthly QA Activities Report to management as described in Section 10.0. All items are addressed at least semi-annually in the QA report to the CEO.

1. Status of or changes to QA Program Plan
2. Status of QA Project Plans, if any
3. Measures of data quality
4. Significant QA problems, accomplishments, and recommendations
5. Results of performance audits
6. Results of system audits
7. Status of QA requirements for contracts
8. Summary of QA training (internal and external QA/QC seminars and courses)

Production Planning & Control SOP 1.1: Logging In Samples

The following steps are completed for all samples as they are received by CompuChem Laboratories. If for any reason a sample requires special handling upon receipt, the Manager of Production Planning and Control is consulted for directions as to the proper handling and documentation of the samples.

- Before opening and while inspecting each sample, each employee is required to wear protective clothing (lab coat, safety glasses and gloves). These items need to be worn at all times when in the marked areas (colored line).

- Each sample container is inspected before opening, making sure that it has not been damaged or opened during shipment. For those clients using padlocks, sealing tape, or custody seals, these items are inspected to make sure that they are intact and this observation is recorded on the chain-of-custody form (Attachment 1). If the custody seals, tapes, or padlocks are broken, one must contact Customer Service (for commercial samples) or the Sample Management Office (for EPA samples) for permission to continue processing the sample.

- Each container is opened under the hood and checked for breakage. The condition of the refrigerant is checked (whether any ice remains or whether the cooling packs are solid), and the temperature is obtained by adhering a calibrated temperature strip to the outside of one representative sample container within each cooler. The temperature is taken and recorded on the Sample Record (Attachment 2) per Production Planning and Control SOP 3.8; Reading and Recording Temperature of Samples.

- The temperature and pH are recorded on the Log-In Sheet. If a temperature or pH variance occurs, a QA Notice is written and associated with the sample (Attachments 3-4).

- Receiving personnel must sign and date all chain-of-custody documentation upon sample receipt and record any discrepancies (sample matrix, for instance) on the chain-of-custody form.

- The Supervisor of Sample Receiving must verify that the Receiving Clerk has signed and dated the chain-of-custody form.

- When a CompuChem SampleSaver is received, the SampleSaver number is recorded on the Log-In Sheet and is entered into the LMS system by number.

- Samples are removed from the shipping container and the sample identification information on the sample bottles is compared to the sample information on the traffic sheets, packing lists, and Chain-of-Custody Form included in the container (Attachments 5-11).* For EPA samples, Form DC-1 is filled out as well, per USEPA SOW. If discrepancies exist, the problem is noted on the Chain-of-Custody Form; the Receiving Clerk notifies Customer Service (for commercial samples) and the Supervisor notifies SMO (for EPA samples).

- Each water VOA is checked for air bubbles and headspace, and noted on the Chain-of-Custody Form as well as the Log-In-Sheet.

- On each complete and correct EPA Chain-of-Custody and Traffic Report the statement "Received in Good Condition" is written or stamped, initialed and dated by the receiving individual.

- On each complete and correct Commercial Chain-of-Custody the statement "Received in Good Condition" is written or stamped, initialed and dated by the receiving individual. "Received in Good Condition" is intended to indicate that the sample or samples were received intact with all associated sample tags (if applicable), custody seals (if applicable), pH for inorganics, and corresponding documentation in order. If there are any discrepancies in the documentation or other problems (such as breakage of the containers or chain-of-custody seals), the exceptions are noted on the appropriate documents, initialed and dated.

- The statement "Received in Good Condition" does not, however, include sample temperature since EPA samples are generally received at temperatures above the recommended 4°C. The temperature is noted on the sample Log-In Sheets and the gray envelope.

- Incoming samples are checked against SMO scheduled receipts (for EPA samples).

* NOTE: For samples associated with NJDEP X-408, an NJDEP059 "Sample Analysis Request Form" will accompany the samples upon receipt (Attachment 12).

- The sample is logged in on the Log-In Sheet Log, noting the following items:

Case number	Temperature
CompuChem sample ID	Client name or order number
Receiving date (RD)	Sampling date (SD)
Analysis codes	Matrix
Volume received	pH (Inorganics Samples Only, see PP&C SOP 3.1)

- For EPA samples, the samples' account data is entered into the marketing section of the CLMS in order to generate the order number and associated requisition numbers. For commercial samples, customer service is contacted to check for the existence of the order and to receive requisitions for analyses. The order is then completed in the CLMS, and the EPA Scheduling Log (Attachment 13) is also completed.

- The sample is entered into the sample receipt portion of the CLMS in order to generate a CompuChem number for each sample. The CompuChem number is filled in on the accessioning log sheet (this completes the log sheet).

- The CompuChem number is a unique, six-digit identity that is generated by the LMS system in numerical sequence. It can be cross-referenced to the Client ID.

- A CompuChem label is generated in numerical sequence, with the CompuChem number.

- The samples are labelled with the CompuChem number by wrapping each sample bottle with its computer-generated CompuChem sample label. Sample labels are color-coded, and are to be rotated with a different color every two-week period by the Supervisor of Receiving or the Supervisor's designee.

- Each Log-In Sheet is reviewed by the Supervisor of Environmental Receiving to ensure information is documented. After review, each log sheet is stamped as reviewed and initialed and dated.

- The labelled samples are transferred to the secured, locked walk-in cooler facility.

- The CompuChem number is listed on the original Chain-of-Custody Form next to the associated client ID when possible.

- The Quiz portion of the CLMS is accessed to produce the worksheets for EPA sample analyses. For EPA samples, the system will generate volatile, semivolatile, and pesticide worksheets. For commercial and inorganic samples, the appropriate worksheets are pulled from the worksheet files; the analysis codes for these samples should have been included with the packing information and confirmed with Customer Service. All laboratory worksheets are distributed to the appropriate Production Planning and Control Planner.

- To produce EPA quality control worksheets for the QC samples associated with a batch of samples, EPA Water or EPA Solid programs of Quiz in the CLMS is accessed and the samples' CompuChem numbers are entered; these worksheets are copied on green paper. To assemble commercial QC worksheets, the appropriate green fraction worksheets are pulled from the trays in the Shipping and Receiving area. Separate QC Sample Records are used to document the analysis of the QC samples associated with a particular system and are put into green QC folders for Report Integration. Included in the commercial folder are the Sample Record (generated by the CLMS), a copy of the order form, and, if necessary, a copy of the Chain-of-Custody Record.

- Commercial file folders are assembled for Report Integration; included in the production sample's folder are the Sample Record, Customer Sample Information Sheet and Chain-of-Custody Record; the QC Sample Record is included in the green Quality Control folder that also goes to Report Integration.

- EPA file folders are assembled for Report Integration; EPA only has the Sample Record in the file folder. A gray envelope contains all materials for the case including: yellow copy of the OTR (Organic Traffic Report), Chain-of-Custody, original air-bill, a copy of the Log-In Sheet, a copy of the EPA Scheduling Log, Custody Tags sealed in plastic bags (if received), and a gray envelope contents sheet (Attachment 9). The white copy of the OTR is returned with a cover sheet to the EPA/SMO (Sample Management Office) (Attachment 10).

- If problems arise concerning received samples, Customer Service is contacted (for commercial samples) or the Technical Management Staff is contacted (for EPA samples).

- Samples are sometimes received from the EPA that should not have been sent. Therefore, they require a transfer. The following steps should be taken:

    1. Fill out a new Chain-of-Custody Form using the information on the sample tags.

    2. Sign the Chain-of-Custody in the first section labeled "Relinquished By."

    3. A Copy of Chain-of-Custody is kept for our records; the original is sent with samples. A copy of the paperwork received with the samples should also be sent.

    4. Notations are made on the Traffic Reports stating samples are being sent to another laboratory.

Samples hand-delivered after business hours should be recorded as follows:

1. The actual date of sample receipt shall be recorded on the Chain-of-Custody (see Note).

2. The date and time of sample receipt are recorded on the TRs as follows:

    a. Organic Traffic Report

    Date and time of receipt in Column F, sample condition upon receipt

    b. Inorganic Traffic Report

    Same as above

The notation "H.D." (Hand-Delivered) or "Received by Common Carrier" should be recorded next to the actual sample receipt time and date. The following calendar day shall be recorded in the block designated for Receipt Date on the Organic/Inorganic Traffic Report.

Samples received after business hours are logged in the same way as samples received during regular business hours. (Page 1 of this SOP details the procedure for logging in samples.)

Note: For New Jersey DEP Chain-of-Custody procedure, see Production Planning and Control SOP 3.2 and the note on page 2 of this SOP.

COMPUCHEM LABORATORIES

CHAIN-OF-CUSTODY RECORD

№ 000933

PROJECT NAME:

PROJECT NUMBER:

SAMPLERS (SIGNATURE)

PRINTED NAME

CLIENT ID (9 CHARACTERS)

1	2	3	4	5	6	7	8	9

#	GC/MS					GC							INORGANICS				OTHER										SAMPLING INFO		REMARKS
No. of Bottles/Vials	624-8240	625-8270	TCL-VOA	TCL-SVOA	Other:	601-8010	602-8020	608-8080	8140	TCL PEST/PCB's	Herbicides	Other:	Metals	Cyanide	TAL Metals	Other:	TOC	TOX	Oil & Grease	Pet Hydro.	Phenols						MATRIX: Water/Soil	DATE	TIME	

Section 1.1
Revision No. 11
Date: June 17, 1991
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ATTACHMENT 1

RELINQUISHED BY	Date/Time	RELINQUISHED BY:	Date/Time	RELINQUISHED BY:	Date/Time
COMPANY NAME		COMPANY NAME:		COMPANY NAME:	
RECEIVED BY	Date/Time	RECEIVED BY:	Date/Time	RECEIVED BY:	Date/Time
COMPANY NAME		COMPANY NAME:		COMPANY NAME:	
RELINQUISHED BY	Date/Time	RELINQUISHED BY	Date/Time	RELINQUISHED BY:	Date/Time
COMPANY NAME		COMPANY NAME		COMPANY NAME:	

SHIPPING INFO

Number of Shipping Containers-

Method of Shipment

Special Handling Requirements

ATTACHMENT 2

Sample Record

Requisition Number: CompuChem Number:
Case: Account Number:
Turnaround: Priority: Due:
Analysis Codes:

Lab Instructions

Receiving Data

Sample Identification: SS Code:
SS Number: Receiving Codes:
Date Received: Time:
Receiving Information:
Containers Received:
Matrix: Temp: Sampling Date(s):

Deliverables

Deliverables Code:

Lab Requirements

Laboratory Completion Date	Repeat Date	
_____	_____	( )
_____	_____	( )
_____	_____	( )
_____	_____	( )

Company Name:

Sample Record

Requisition Number: CompuChem Number:
Case: Account Number:

FOOTNOTES:

Applicable QA Notices:

Company Name:

QUALITY ASSURANCE NOTICE

CompuChem# _____
Sample ID _____
Case# _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was ____, the required pH level is ____.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

Preserve In-House _____
Analyze – Qualify with Notice _____
Dispose – Client will Resample _____
Subcontract Lab to Preserve _____

Supervisor Signature _____
Date _____

QANR2
900130

QUALITY ASSURANCE NOTICE

CompuChem#
Sample ID
Case#
Type of Analysis
Receipt Date

The required temperature for Environmental samples requiring Organic/Inorganic Analysis is 4C (+/-2C). The temperature on the sample above was ____.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

Analyze - Qualify with Notice
Dispose - Client will Resample

Supervisor Signature
Date

QANR3
900130

COMPUCHEM LABORATORIES
ENVIRONMENTAL RECEIVING LOG

RECEIVED ON:		# OF PAGES ___ OF ___
CLIENT:	ORDER#	CASE I'D
DELIVERED BY:	FREIGHT #'s	TAT ___ C-OF-C Y or N
SUBCONTRACT#	SS#	

#	REMARKS	REQ	SAMPLE ID	CC	SO	TEMP	Su	Ch	pH	ANAL CODES	MX	VOLUME
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

LOGGED-IN BY: RECEIVED BY: REVIEWED:

ATTACHMENT 6

COMPUCHEM LABORATORIES
ENVIRONMENTAL RECEIVING LOG

RECEIVED ON: | # OF PAGES OF
CASE # | ORG/INORG: | ORDER# | CASE I'D
DELIVERED BY: | FREIGHT #'s | TAT C-OF-C Y or N
ACCOUNT #: | CONTRACT # | REGION: | $ AMT: | TAGS Y or N

#	REMARKS	REQ	SAMPLE ID	CC#	#SO	TEMP	SU	CN	pH	ANAL CODES	MX	VOLUME
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

LOGGED-IN BY: RECEIVED BY: REVIEWED:

COMPUCHEM LABORATORIES

date shipped to consignee:_____ date received from consignee: _____

number of sample: _____

consignee name: _____

address: _____

_____

_____

DO NOT REMOVE: FOR COMPUCHEM USE ONLY

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION

At the end of the sampling period, it is vital to ship the sample via express transportation. To insure proper follow-up and prompt analysis, please call 1/800-334-8525 and provide us with the following information:

1. Date Shipped
2. Time Shipped
3. Freight Carrier
4. Freight Bill of Lading Number

Sampling Period

From:

Date _____

Time _____

To:

Date _____

Time _____

Company _____

Address _____

City & State _____ Zip Code _____

Sample Name/Number _____

Return this form in the envelope provided and return with the SAMPLESAVER.

Thank you.

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION.

COMPUCHEM

CONTENTS OF FOLDER

DATE: _____

EPA: ( ) ORGANIC ( ) INORG COMMERCIAL: ( )

CASE #: _____ CLIENT: _____

NO. OF TRAFFIC RPTS: _____ ORDER#: _____

#	ITEM	Y	N	NA	COMMENTS
1	CHAIN-OF-CUSTODY	( )	( )	( )	
2	AIRBILL	( )	( )	( )	
3	DAILY LOG SHEET	( )	( )	( )	
4	TRAFFIC REPORT	( )	( )	( )	
5	TAGS	( )	( )	( )	
6	SMO COVER LETTER	( )	( )	( )	
7	SCHEDULING LOG	( )	( )	( )	

PROBLEMS/COMMENTS:

SAMPLE RECEIVING:

DOCUMENTATION CONTROL:

COMPUCHEM LABORATORIES

SAMPLE DELIVERY GROUP (SD
TRAFFIC REPORT (TR) COVER S

Lab Name: _____ **Contract No.: 68-01-**_____

Lab Code: _____ **Case No.:** _____ **SAS No.:** _____-___

Full Sample Analysis Price in Contract: $_____

SDG No./First Sample in SDG: _____ **Sample Receipt Date:** _____
(Lowest EPA Sample Number in first shipment of samples received under SDG) (MM/DD/YY)

Last Sample in SDG: _____ **Sample Receipt Date:** _____
(Highest EPA Sample Number in last shipment of samples received under SDG) (MM/DD/YY)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1	_____	11	_____
2	_____	12	_____
3	_____	13	_____
4	_____	14	_____
5	_____	15	_____
6	_____	16	_____
7	_____	17	_____
8	_____	18	_____
9	_____	19	_____
10	_____	20	_____

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

_____ _____ _____

Sample Custodian Date

SAMPLE LOG-IN SHEET

Lab Name: _____ Page ____ of ____

Received By (Print Name): _____ Log-in Date: _____

Received By (Signature): _____

Case Number: _____

Sample Delivery Group No.: _____

SAS Number: _____

REMARKS:

1. Custody Seal(s) Present/Absent* Intact/Broken
2. Custody Seal Nos.: _____ _____
3. Chain-of-Custody Records Present/Absent*
4. Traffic Reports or Packing List Present/Absent*
   Airbill Airbill/Sticker Present/Absent*
6. Airbill No.: _____ _____
7. Sample Tags Present/Absent*
   Sample Tag Numbers Listed/Not Listed on Chain-of-Custody
8. Sample Condition: Intact/Broken*/Leaking
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*
10. Date Received at Lab: _____
11. Time Received: _____

Sample Transfer

Fraction: _____ _____

Area #: _____ _____

By: _____ _____

_____ _____

	CORRESPONDING		
EPA SAMPLE #	SAMPLE TAG #	ASSIGNED LAB #	REMARKS: CONDITION OF SAMPLE SHIPMENT, ETC.

* Contact SMO and attach record of resolution

Reviewed By: _____

Date: _____

Logbook No.: _____

Logbook Page No: _____

Form DEP-058
10/86

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTE

SAMPLE ANALYSIS REQUEST FORM

SAMPLE LOCATION

NAME OF FACILITY/ COMPANY/PERSON	CODE	MAILING ADDRESS
SAMPLE LOCATION		

SAMPLE COLLECTOR

SAMPLER(S) Name	DIVISION		
Name	CONTRACT #	Task #	Report Format

SAMPLE IDENTIFICATION

NJDEP SAMPLE NO.	SAMPLING DATE	ABSCISSA (Longitude)
SAMPLE POINT ID	TIME (MIL) SAMPLING BEGAN	ORDINATE (Latitude)
SAMPLE SOURCE CODE	TIME (MIL) SAMPLING FINISHED	ELEVATION
REFERENCE POINT		

SAMPLING PROCESS

COLLECTION METHOD	Code	Other	SAMPLE TYPE	Code	Other
TYPE OF SAMPLER	Code	Other	SAMPLE MATRIX	Code	Other
DESCRIPTION					

FIELD MEASUREMENTS

SAMPLE TEMP. °C	PARAMETER	PARAMETER	PARAMETER
AIR TEMP. °C	VALUE	VALUE	VALUE
WEATHER			

CHAIN-OF-CUSTODY IMPLEMENTED ☐ YES ☐ NO | PRESERVATION CODE(S) ___ ___ ___ ___ ___

REQUESTED ANALYSIS(ES)

PARAMETERS (by Compound or Group of Compounds)	CONTAINER NUMBER	DETECTION LIMIT (digits/units)	EMERG.	NON-EMERG. (Check One)

ANALYSIS(ES) AUTHORIZED BY | TITLE

ANALYZING LABORATORY

CONTRACT LAB NAME	PERSON ACCEPTING SAMPLE	TITLE
LAB ID #	LAB CONTROL #	DATE	TIME (MIL)

COMPUCHEM LABORATORIES

EPA SCHEDULING LOG

WEEK ENDING SATURDAY:

CASE#: ( ) NEW CASE ( ) OLD CASE ( ) COMPLETED CASE

REGION: TURNAROUND TIME:

ACCOUNT #: CONTRACT #: CONTRACT $

****

QUANITY RECEIVED # OF SAMPLES

WATERS	
SEDIMENTS/SOILS	
OTHER	

CONCENTRATION: ( ) LOW ( ) MEDIUM ( ) HIGH

ORDER#:	
AIRBILL#:	

****

PROBLEMS/COMMENTS:

SMO CONTACTED AT (TIME) SPOKE TO:

RESOLUTION FROM SMO:

Documentation Form For:

Revising or Creating Standard Operating Procedures (SOPs): Including Designated Personnel Responsibilities

✓ Revised Procedure _____ New Procedure ✓ Procedure Attached

PPC SOP 1.1: Logging In Samples — 6-17-91
Procedure Area, Title, and SOP Number — Effective Date

Michael Grasso — 6-17-91
Procedure Prepared By — Date

Michael Grasso — 6-17-91
Procedure Read, Understood, and Approved By
Appropriate Laboratory Station Manager — Date

[signature] — 6-18-91
Procedure Read, Understood, and Approved By
Quality Assurance Representative — Date

This procedure(s) meets the requirements as set forth in the following References for Approved Methods:

_____

_____

_____

_____

These procedures describe how tasks are performed in this specific area. If a question arises concerning the proper procedure to follow for an activity in this area, these SOPs should be consulted to resolve the question. Also, these SOPs are a valuable source of material for training purposes.

After the manager of this area believes the person responsible for these tasks has mastered these SOPs, both the manager and the employee should sign and date this form, assuring that these SOPs are understood and will be followed in the daily operations of CompuChem Laboratories. Please forward a copy of this revised or created SOP and a completed form to Quality Assurance.

Employee's name: _____ Date: _____

Employee's title: _____

Employee's name: _____ Date: _____

Employee's title: _____

Manager's name: _____ Date: _____

Manager's title: _____

APPENDIX F

Drinking Water Requirements

APPENDIX F

Drinking Water Requirements

Samples identified by the client as "Drinking Water Samples" (i.e., for drinking water compliance monitoring) require certain special handling and reporting procedures, but are otherwise handled by the Computerized Laboratory Management System (CLMS) in much the same way as non-compliance samples.

The Sales Representatives in the Marketing Department are responsible for placing the order in the CLMS, ensuring that the appropriate analysis codes are chosen. Only analysis codes describing EPA-approved drinking water methods may be used. The tables on the following pages identify the particular methodologies utilized in processing drinking water samples.

For compliance monitoring in North Carolina, following the "Rules Governing Public Water Supplies" (amended February 1, 1987), all certified commercial laboratories are required to report results of analyses to both the Public Water Supply Branch and the supplier of water (client). The rules specify the particular reporting forms to be used and the time period in which reports are to be submitted.

In evaluating drinking water sample data, the QC criteria applied are as specified in the referenced method. Where unspecified, CompuChem® employs those criteria outlined in the Federal Register (October 26, 1984 600-series methods) for "Water and Wastewater", presented in Section 9.5 of the QA Plan. Once a database of sufficient size is generated, control limits for precision and accuracy will be generated based on historical data for aqueous sample analyses.

In order to continue providing analytical services for compliance monitoring, CompuChem® must maintain certification through the various drinking/potable water certifying agencies. The North Carolina Department of Human Resources (NCDHR), Division of Health Services, regulates certifications, performance evaluations and annual on-site laboratory inspections for these services in North Carolina. CompuChem® also maintains drinking water certifications in a number of other states, many of which accept reciprocal certification through the NCDHR.

METHODS USED BY COMPUCHEM®
FOR POTABLE WATER ANALYSIS

Volatile Organic Contaminants/THMS	Method Used *
Bromobenzene	524.1
Bromochloromethane	524.1
Bromdichloromethane	524.1
Bromoform	524.1
Bromomethane	524.1
sec-Butylbenzene	524.1
tert-Butylbenzene	524.1
Carbon tetrachloride	524.1
Chlorobenzene	524.1
Chlorodibromethane	524.1
Chloroethane	524.1
Chloroform	524.1
Chloromethane	524.1
o-Chlorotoluene	524.1
p-Chlorotoluene	524.1
1,2-Dibromo3-Chloropropane	504,524.1
Dibromethane	524.1
o-Dichlorobenzene	524.1
m-Dichlorobenzene	524.1
p-Dichlorobenzene	524.1
Dichlorodifluoromethane	524.1
1,1-Dichloroethane	524.1
1,2-Dichloroethane	524.1
1,1-Dichloroethylene	524.1
cis-1,2-Dichloroethylene	524.1
trans-1,2-Dichloroethylene	524.1
Dichloromethane	524.1
1,2-Dichloropropane	524.1
1,3-Dichloropropane	524.1
2,2-Dichloropropane	524.1
1,1-Dichloropropane	524.1
1,3-Dichloropropane	524.1
Ethylbenzene	524.1
Ethylenedibromide	504,524.1
Flurotrichloromethane	524.1
Hexachlorobutadiene	524.1
Isopropylbenzene	524.1

* "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", September, 1986, EMSL-CI, U.S.EPA Cincinnati, Ohio 45268.

METHODS USED BY COMPUCHEM®
FOR POTABLE WATER ANALYSIS
(continued)

Volatile Organic Contaminants/THMS	Method Used *
n-Propylbenzene	524.1
Styrene	524.1
1,1,1,2-Tetrachloroethane	524.1
1,1,2,2-Tetrachloroethane	524.1
Tetrachloroethylene	524.1
1,1,1,-Trichloroethane	524.1
Trichloroethylene	524.1
Toluene	524.1
1,1,2-Trichloroethane	524.1
1,2,3-Trichloropropane	524.1
Vinyl chloride	524.1
o-Xylene	524.1
m-Xylene	524.1
p-Xylene	524.1
Chlorodane	608
Polychlorinated Biphenyls	608

Inorganic Contaminants	Method Used **
Iron	236.2
Manganese	243.2
Arsenic	206.2
Barium	208.2
Cadmium	213.2
Chromium	218.2
Fluoride	340.2
Lead	239.2
Mercury	245.1
pH	150.1
Selenium	270.2
Silver	272.2
Sodium	273.2

* "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", September, 1986, EMSL-CI, U.S.EPA Cincinnati, Ohio 45268.

** "Methods for Chemical Analysis of Water and Wastes," EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, 45268 (EPA-600/4-79-020), March 1979. Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268. For approved analytical procedures for metals, the technique applicable to total metals must be used.

APPROVED METHODOLOGY FOR ORGANIC CONTAMINANTS

Additional Organic Contaminants	Method Used
Chlorinated Hydrocarbons	1
Endrin	1
Lindane	1
Methoxychlor	1
Toxaphene	1
Chlorophenoxy, Acids	2
2,4,-D	2
2,4,5-T	2

1 : "Methods for Organochlorine Pesticides and Chlorophenoxy Acid Herbicides in Drinking Water and Raw Source Water," Available from ORD Publications, CERI, EPA, Cincinnati, Ohio 45268. (pp. 1-19)

2 : Ibid. (pp. 20-35)

10.0 Corrective Action

10.1 Introduction

Generally, there are two types of corrective actions that may be required when data quality falls below specified limits. The first type, and the simplest to implement and document, is corrective action required because routine data quality assessments are out-of-control. Surrogate and spike standard recoveries, relative percent differences between duplicates, internal standard response variations, and unacceptable blank contamination are some of these assessments in the first category. These are all performed on a sample-by-sample and/or batch basis, and corrective action is limited to evaluating the data with respect to SOP criteria, and accepting or rejecting the sample/batch. The decision that is made is clearly indicated on analytical worksheets, and unless a trend is observed during the course of data validation, additional corrective action or documentation is not necessary.

The second type of corrective action is that required when other, more global QC/QA assessments, are made. The assessments might typically indicate systematic deficiences or those affecting data useability for more than one batch (i.e., glassware contamination checks, standards preparation errors, etc.). In most cases, assessments of this nature are made by reviewing peripheral QC/QA documentation, observing procedures for comparison with SOPs or GLPs, or receiving feedback from data reviewers, management or those external to the organization (clients, auditors).

The following sections describe the QA reporting and feedback channels designed to ensure that early and effective corrective action is taken in such instances.

In many cases, depending on the nature of the deficiency and the urgency for remedial action, a Corrective Action Report (following this section) will be completed. The report serves to document the deficiency, the required corrective action, and accountability for the action.

For observations made over longer periods of time, the QA Department issues formal summary reports to management on a monthly or quarterly basis. Following is a brief discussion of the types of reports issued to management to assess the overall effectiveness of the QA Program and to reinforce the application of Good Laboratory Practices (GLPs).

CORRECTIVE ACTION REPORT

DATE:_____

PROBLEM / DEFICIENCY:

_____

_____

_____

_____

_____

IDENTIFIED BY: _____

REFERRED TO: _____ (QA)

CORRECTIVE ACTION TO BE TAKEN: TARGET DATE:_____

_____

_____

_____

_____

_____

_____

FOLLOW-UP AUDIT FINDINGS:

_____

_____

_____

_____

RESOLVED? _____ DATE: _____

SOP REQUIRED TO BE WRITTEN/MODIFIED? YES [ ] NO [ ] TARGET DATE:_____

This form to be filed with the Quality Assurance Clerk for permanent record.

10.2 Routine QC Check Reports

The following routine quality control checks (also discussed in section 9.2 of the QA Plan) are performed to verify that samples are not contaminated during transportation, preparation, analysis or storage, and that standards prepared internally are traceable to certified sources.

- -- Vendor-Supplied Glassware Checks
- -- Glassware Decontamination Checks
- -- Water Purification Systems Checks
- -- Glassware Storage Cabinet Checks
- -- Refrigerated Storage Systems Checks
- -- Reagent Purity Checks
- -- Standards Prepartion and Traceability Checks

The criteria for these QC checks and corrective action steps are detailed in the QA SOP Manual. Results are tabulated and/or plotted on control charts, and records reviewed by the QA staff. A series of quarterly reports to management summarize this information and the status of these programs.

10.3 Monthly QA Activity Reports

These reports are produced by all members of the QA staff, and summarize key QA activities during the previous month. The reports are distributed to the Director of QA, and are provided as an attachment and referenced in the Director's report to the CEO, the Executive Staff and senior laboratory management.

Included in these reports is a summary of significant quality problems observed during the period, and the corrective actions taken to remove deficiencies. The report stresses proactive measures that are being taken to improve quality or ensure compliance with QA program requirements.

Laboratory management uses the report to quantitatively measure monthly performance in terms of the number of samples processed, the frequency of repeated sample analyses due to unacceptable QC performance, and the cause of the unacceptable performance. These data are all presented in tables, Pareto control charts or attribute control charts, based on the characterization of each analysis in the Computerized Laboratory Management System (CLMS) using a system of analytical "condition codes."

The Condition Code System is used to monitor sources of data failures. Condition code definitions are provided in an SOP to data generators and reviewers who are responsible for assigning the appropriate code to each analysis (see Appendix D). Each two-letter code is used to characterize the cause of a sample failure or the final status of the data package prior to release to the client.

Various computer programs may be used to sort condition code data according to sample matrix and method. This system is used to pinpoint sources of error, provide feedback to management, reinforce good laboratory practices, and document laboratory performance over time. The QA staff also note in the Monthly QA Activities Report any corrective actions taken or necessary procedural changes, based on the application of condition codes.

Other items included in this report are:

-- Summary of any changes in certification/accreditation status

-- Involvements in resolution of quality issues with clients or agencies

-- QA organizational changes

-- Notice of the distribution of revised documents controlled by the QA Department (i.e., SOPs, QA Plan)

-- Training and safety issues, if not already covered in audit reports during the period

-- Performance of subcontractor laboratories (also communicated in separate, detailed subcontractor audit report to management)

-- Positive feedback for acceptable performance on interlaboratory or intralaboratory tests or successful completion of audits.

10.4 Laboratory Performance Reports

This quarterly report presents a statistical and graphical summary of the laboratory's performance on batch-associated quality control samples analyzed over the period. Included are tables, Shewhart control charts and I-charts (for individual data points) for all surrogate and spike standard recoveries. Additionally, a monthly report to the Director of QA presents control charts and tables for all Laboratory Control Sample (Blank Spike) and Blank recoveries. The charts and tables are used primarily to document historical performance, update recovery control limits, and monitor long-range trends that might not be apparent to data reviewers evaluating data on a sample/batch basis.

10.5 Laboratory Audit Reports

Quarterly audit reports are written by a member of the QA staff and distributed to management, and summarize the results of internal laboratory Performance Audits, Systems Audits and Security/Access Audits. When external auditors are involved in Performance or System Audits, a report is written within the next week by the QA staff member coordinating the audit. The report, summarizing audit results as discussed in the debriefing as well as other observations, is distributed to the CEO and senior lab management. The report includes corrective actions required as a result of the audit, and a schedule for implementation. A follow-up audit, usually within three weeks of the distribution of this report, is conducted to verify that corrective actions have been implemented.

Performance Audits

Performance Audits are checks made by a QA staff member or other independent auditors to evaluate the quality of the data produced by the analytical system. These audits are performed independent of an in addition to routine quality control checks, and reflect as closely as possible lab performance under normal operating conditions.

These audits involve the review of approximately 10% of all analytical data reports generated by the lab for calculation and data validation procedures, and overall data quality. Errors observed during the audit are characterized as "critical" or "correctable" and tabulated. If necessary, based on audit findings, an amended data report may be sent to the customer. Following this section is a copy of the QA Audit Summary used by auditors to tabulate the data

for summary into the Quarterly Performance Audit report. A thorough discussion of these audits is included in the QA SOPs. The reports are used by laboratory managers to provide feedback to staff members and establish goals for improved performance.

A number in interlaboratory and intralaboratory tests are conducted routinely at CompuChem®, and the results are included in individual Performance Audit reports specific to each test. When new methods are available to the laboratory or new personnel are being trained, Laboratory Proficiency Tests are performed. These tests consist of quadruplicate blank spikes, containing a full complement of tests parameters to be analyzed by the method. The replicate results are analyzed by a QA staff member, who generates a summary report to the Director of QA. This report includes the standard deviation and mean recovery for each of the replicate parameters, and the data are used to statistically validate method and/or personnel proficiency. For a thorough discussion of the method validation procedures used, refer to Appendix A of the QA Plan.

On a quarterly basis, blind intralaboratory check samples are introduced into the system by the QA Department. Parameters and methods are chosen for these studies based upon independent (interlaboratory) tests from certifying agencies (including the U.S. EPA and various state agencies), Laboratory Proficiency Test results, Method Validation studies, or results from routine batch-related QC samples. The existence of these check samples in the system is known only to those personnel involved in preparing the samples and scheduling the analytical requirements into the CLMS. A thorough report, detailing the entire data generation and support functions, is completed by the QA staff and reviewed by

the Director of QA before distribution to the CEO and senior laboratory management.

CompuChem® also participates in a number of external, interlaboratory performance studies. These are required as part of various agencies' certification/accreditation programs. As a member of the USEPA's Contract Laboratory Program (CLP), the laboratory is required to successfully analyze quarterly, blind proficiency samples for both organic and inorganic parameters. The CLP program also requires an annual on-site inspection by principals from the USEPA (and their contracted agents). These audits generally follow the same format described below, Systems Audits.

CompuChem® also participates in a number of state certification programs, including those for North Carolina, New Jersey, New York and Florida. All of these programs require the laboratory to submit to annual on-site inspections in order to maintain certification to perform testing on samples originating in the state. All states also require successful performance on interlaboratory check samples, submitted at least annually, though some reciprocity with the two NC programs (one for drinking water and one for wastewater certification) and USEPA-CLP is allowed under certain circumstances.

Several states utilize the laboratory's performance on the annual Water Supply (WS) and Water Pollution (WP) proficiency testing series, orginating out of the EPA Environmental Monitoring and Support Laboratory's performance on all interlaboratory and intralaboratory check samples, tabulated by parameter and method, so negative performance trends can be readily pinpointed.

System Audits

A System Audit is an on-site inspection and review of the QA Program for the total laboratory. While Performance Audits are a quantitative appraisal, System Audits are for the most part qualitative in nature. The System Audit may be either scheduled or unannounced before it is conducted, but occurs routinely on at least a quarterly basis. The auditor reviews the laboratories' SOPs to verify compliance with procedures and activities actually in place. Personnel and facilities are also evaluated during the System Audit. The auditor is required to investigate anything which seems in conflict with the QA Plan, the laboratory or QA SOPs, or Good Laboratory Practices.

If deficiencies are observed during a Performance Audit, and if deemed necessary, the QA Department initiates a System Audit. The audit emphasizes the actions necessary to correct deficiencies noted in the Performance Audit. A Corrective Action Report is completed, detailing all remedial actions taken, and reviewed by the Director of QA. The report must indicate the proposed implementation date and the individual(s) responsible for the action.

Many of the objectives of a routine System Audit are similar to those a client or independent auditor would hope to accomplish during an On-Site Laboratory Evaluation and Data Audit. These goals include ensuring the following:

1. The quality control, including necessary corrective actions , are being applied
2. Adequate facilities and equipment are available to perform the client's required scope-of-work
3. The personnel are qualified to perform the assigned tasks
4. Complete documentation is available, including sample chain-of-custody

5. Proper analytical methodology is being applied

6. Acceptable data handling techniques are being used

7. Corrective actions identified in any previous on-site visits have been implemented, and

8. The Laboratory Management continues to demonstrate a commitment to quality.

These objectives may be documented by completing an EPA-approved Laboratory Evaluation Checklist. In response to System Audits, any corrective actions taken are noted with reference to the auditor's deficiency report and the lab's Standard Operating Procedures.

	SAMPLE NUMBER	FRACTION	REJECT LOCATION/CAUSE						COMMENTS	SUMMARY corr* failure	SUMMARY crit** failure	SUMMARY status
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												
21												
22												
23												
24												
25												
26												
27												
28												

*correctable failures

**critical failures

DATE:

QA AUDIT CODES

Missing/Incorrect:

CAM/I	Calculations missing/incorrect
CCM/I	Condition code missing/incorrect
DFM/I	Data footnote missing/incorrect
CFM/I	Correction factor missing/incorrect
DWI	Dry weight/percent moisture incorrect
FNI	Filename incorrect
FFM/I	Form 4 missing/incorrect
LSM/I	Library search missing/incorrect
QNM/I	QA Notice missing/incorrect
RRM/I	Reportable run missing/incorrect
SPM/I	Spectrum missing/incorrect
SRM/I	Sample receiving information missing/incorrect
SSM/I	Surrogate Summary Form missing/incorrect
STM/I	Standard package missing/incorrect
TFM/I	Tuning Form missing/incorrect
UNM/I	Units missing/incorrect
WSM/I	Worksheet missing/incorrect/incomplete
OAM/I	OADS missing/incorrect/incomplete

Qualitative/Quantitative Errors:

HNR	Hit not reported, but should have been
HRE	Hit reported in error, should not have been reported
HAI	Hit amount reported incorrectly
CFN	Correction factor not applied to hit
SFI	Significant figures (or rounding off) incorrect
TRE	Transcription error

Miscellaneous Errors:

ISF	Internal standard area monitor indicates failure
ODI	OWA date or time incorrect
RNL	RIC not labeled
SOL	Surrogate(s) actually outside limits
WOU	Whiteout used on documents (deliverables)
NSO	Not signed off
CNI	Change not initialed

Condition Code Applications:

CS	Carryover suspected
CT	Contamination evident
RU	Repeated unnecessarily
SF	Spikes failed
UN	Unacceptable, not needed

6.4 Instrument Maintenance

Analytical instruments are maintained by experts employed by CompuChem® on a full-time basis. Preventative maintenance as well as major instrument repairs can be accomplished on-site. An extensive in-house stock of spare parts allows for rapid repair. CompuChem® maintains service agreements with instrument manufacturers to further assure the operational viability of all in-house equipment.

The operational condition of instruments is one of the keys to successful completion of analytical tasks. This requirement is further magnified by the necessity to complete large programmatic requirements in a limited period of time. CompuChem's commitment to instrument maintenance assures clients that equipment will be available to generate the required data.

In discussing instrument maintenance services at CompuChem®, a distinction between GC/MS instruments and other hardware is required. In the case of the GC/MS instrumentation, CompuChem® staff have full maintenance and repair responsibility. These staff have been trained by the instrument manufacturer and are fully qualified to perform the required work. For other instruments, we have service contracts for periodic maintenance visits by the vendor, although maintenance personnel do assess whether repairs can be made in-house before outside vendors are called.

All GC/MS instrument repair logs and instrument service records are maintained in individual instrument files in the instrument repair shop.

EXAMPLE 1

COMPUCHEM SERVICE REPORT R-1234 A

INSTRUMENT NO 1 2 DATE 0 7 2 3 8 6 TIME 1 1 5 1 OPERATOR: Susan Bass

GC oven not heating

START DATE	0 7	2 3	8 6	END DATE	0 7	2 3	8 6	RESPONSE TIME	5	S/E Lee Gregory				
START TIME		1 0	1 5	END TIME		1 0	5 7	TOTAL DOWNTIME	1 2	JOB COMPLETE? YES	X	NO		

ASSEMBLY NO.	REV LEVEL	REPAIR CODE	FAILURE CODE	COMPONENT DESIGNATOR	PART NO.	DESCRIPTION	QTY	COST
7 0 1 7 8 9 1 4 3 2 0 1	0 A	0 4	0 6	F 4	3 A G 4 A	Fuse	1	1 35

COMMENTS: Oven not heating, 24 volt relay not engaging found fuse F4 blown in power supply. Replaced fuse F4, checked +24 volts, checked operation of GC, gave back to operator L.G.

OPERATOR ACCEPTANCE: DATE 0 7 2 3 8 6 TIME 1 1 0 0 REV07298216

EXAMPLE 1 (CONTINUED)

INSTRUCTIONS

1. INSTRUMENT NO. - LIST AS 05 FOR OWA05, 12 for OWA12, ETC. THE 4021 GC/MS/DS IS INSTRUMENT 00. ALL STANDALONE DATA SYSTEMS ARE INSTRUMENT 99.
2. DATE & TIME - ENTER DATE AS MM/DD/YY; AUGUST 28, 1985 IS 08/28/85. ENTER TIME BY 24-HOUR CLOCK. 9:25AM IS 0925 AND 9:25PM IS 2125. THE TIME AND DATE SHOULD BE WHEN A PROBLEM IS DISCOVERED AND REPORTED VIA THIS FORM.
3. OPERATOR - WHO YOU ARE.
4. PROBLEM CODE & DESCRIPTION - USE THE 3 DIGIT PROBLEM CODE THAT MOST APPROPRIATELY DESCRIBES YOUR PROBLEM. PLEASE DETAIL THE PROBLEM AS FULLY AS YOU CAN.
5. USE BLACK INK ONLY & WRITE OR PRINT LEGIBLY.

PROBLEM CODES (CAUSE & EFFECT)	
P.M.	000
CANNOT MEET TUNE	001
IDOS ERRORS - LIST AND FULLY DESCRIBE WHAT THE DATA SYSTEM WAS DOING	002
POOR SENSITIVITY	003
DRIFTING RET. TIMES	004
NO SPECTRA OR NO MS RESPONSE	005
SOFTWARE ANOMALIES	006
GAS CHROM.	007
DISC DRIVE	008
PRINTER	009
PURGE & TRAP	010
VACUUM FAULT	011
AIR LEAKS	012
TERMINAL	013
DATA SYSTEM	014
CANNOT BOOT	015
UNKNOWN	016

REPAIR ACTION CODES	
PIRATE PARTS	100
ADJUSTMENT - ELEC	102
ADJUSTMENT - MECH.	104
REPLACED ASSY.	106
RETURNED TO VENDOR REPAIR	108
RETURNED TO VENDOR WARRANTY	110
REQUESTED IN-HOUSE VENDOR SERVICE	112
WAITING FOR PARTS (NOTE P.O. #)	114
CLEANED SEPARATOR	116
CLEANED MASS FILTER	118
CLEANED SOURCE	120
REPLACE PART	122
REPAIR IN-HOUSE	124
UNABLE TO REPRODUCE	126

FAILURE ANALYSIS CODES	
UNKNOWN	200
MISCELLANEOUS	202
OPERATOR ERROR	204
SOFTWARE	206
HEADCRASH	208
MECH. DEFECT	210
OUT OF ADJUSTMENT	29*
INTERMITTANT	29*
EXCESSIVE NOISE	29*
EXCESSIVE WEAR	212
SHORTED COMPONENT	214
OPEN COMPONENT	216
FAULTY CRIMP	218
POOR CONTACT	220
POOR SOLDER JOINT	222
DIRTY/DUSTY	224
LEAKING	226
REPLACE * WITH	
1 = ELECTRICAL 2 = MECHANICAL 3 = VACUUM 4 = SOFTWARE	

REV07298216OO

PREVENTIVE MAINTENANCE - 3 MONTH INTERVAL

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE*

ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	SERVICE INTERVAL						SERVICE INTERNAL	PROCEDURE
		daily	bi-monthly	3 months	6 months	yearly	as required		
Sigma 3 GC								NOTE: Applicable procedures are presented in the Finnigan Operator Manual(s), unless otherwise specified.	
1. Line fuses for the GC	Inactive GC, blown fuse						X	replace fuse	
2. Injector for packed columns							X		
3. Splitless injection for capillary columns							X		
4. Injector septum in the GC	obstruction, leaks	X					X	clean, inspect or	
5. Carrier gas connections/couplings	leakage						X	replace as required	
6. Carrier gas filter in the GC	replace when new gas cylinder is installed						X		
7. Filter, flow controller	dirty filter			X				replace filter clean	
8. Capillary column							X	inspect or replace as	
9. Packed column (glass type)	excessive usage, leaks at injection and interface port of the zone-heating block						X	needed	
10. Packed column (metal type)							X		
11. Detector port to GC/MS interface							X		
12. GC cool down fan		X		X				inspect and/ or replace	

*These maintenance procedures meet or exceed Finnigan's recommended preventive maintenance checks and services.

ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	SERVICE INTERVAL						SERVICE INTERNAL	PROCEDURE
		daily	bi-monthly	3 months	6 months	yearly	as required		
Mass Spectrometer									
1. Glass jet separator	obstruction or glass breakage						X	clean or replace	
2. Glass jet separator ferrules							X	replace	
3. Mass analyzer head assembly (in the vacuum manifold)	gross leaks, presistent pressure due to degasing of trapped gases in the vacuum system						X	Inspect	
*magnet wall flange assy	leakage, faulty CAL gas pressure (see Pirani gauge) faulty switch						X	Inspect	
*CAL gas valve assy							X	Inspect	
*vent valve assy							X	Inspect	
*water flow sensor switch							X		
4. Quadrupole mass analyzer				X				Inspect and/ or replace	
5. Electron multiplier							X		
6. Alcatel vacuum pumps (2)				X				purge weekly and replace oil	
7. Pfeiffer turbo pump	dirty oil								
8. Balzer turbo pump				X					Balzer Manual pg...23
9. Vacuum system filter/drier	excessive use, dirty filter						X	clean & inspect	
10. Ion Source				X				replace clean, inspect or replace as required	
*Ion source filament assy	lack of sensitivity, faulty peak shape, no autotune	Service concurrently with every filament assembly replacement							
*collector									
*lens									
*aperture									
*Ion volume									
GC/MS Interface Oven									
1. Capillary interface tubing	plugged						X	clean, inspect and/or replace	
2. Separator divert fitting				X			X		
3. Vacuum divert valve				X			X		
Power Module									
1. MS power supply				X			X	measure & verify PCB	
2. Turbo power supply				X			X		
Card Cage Module									
1. Air filter at bottom of cage	dirty filter, obstruction of air flow			X				clean and/or replace	
2. Fan	burned out fan			X			X		
3. Signal cable on Digital I/O PCB							X	Inspect for secure fit	

REPAIR - PREVENTIVE MAINTENANCE CHECKS AND SERVICES GUIDE (Cont.)

ITEMS TO BE INSPECTED	PROBABLE SYMPTOM	SERVICE INTERVAL						SERVICE INTERNAL	PROCEDURE
		daily	bi-monthly	3 months	6 months	yearly	as required		
Nova Computer								Inspect and/or replace	
1. Fan	faulty fan rotation			X					
Perkin-Elmer Disk Drive									
1. Output signal				X				check and verify	
2. Adjustable DC voltages (+5V, +13V, -13V)				X					
3. Brushes								clean and/ or replace	
4. Positioner carriage guide rails					X			clean and inspect	
5. Spindle chuck and cone					X			clean and inspect	P/ECEM Manual
6. Read-write heads				X				inspect and repair	P/ECEM Manual
7. Fixed disk				X					
Air filter						X			P/ECEM Manual
*prefilter				X				replace	
*main filter				X				replace	
9. Blower ground brush						X		replace	
10. Spindle ground brush						X		replace	
11. Blower drive belt								replace	P/ECEM Manual

COMPUCHEM LABORATORIES

PROJECTED PREVENTIVE MAINTENANCE SCHE:

FINNIGAN GAS CHROMATOGRAPH / MASS SPECTROMETER

JANUARY 1990 TO JUNE 1990

UNIT #	NORMAL ROUTINE	DUE FROM PREVIOUS ***	JAN	FEB	MAR	APR	MAY	JUN
01	04-08-12					DUE		
02	03-07-11				DUE			
03	01-05-09		DUE				DUE	
04	03-07-11				DUE			
05	03-07-11				DUE			
06	01-05-09		DUE				DUE	
07	02-06-10			DUE				DUE
08	02-06-10			DUE				DUE
09	04-08-12					DUE		
10	04-08-12					DUE		
11	02-06-10			DUE				DUE
12	02-06-10			DUE				DUE
13	01-05-09		DUE				DUE	
14	01-05-09		DUE				DUE	
15	04-08-12					DUE		
16	01-05-09		DUE				DUE	
17								
18	03-07-11				DUE			
19	03-07-11				DUE			
20	03-07-11				DUE			
21	02-06-10			DUE				DUE
22	04-08-12					DUE		
23	02-06-10			DUE				DUE

APPENDIX E

GENERAL STANDARD OPERATING PROCEDURES FOR WARZYN, INC.

Revision 8-18-87

WARZYN QUALITY CONTROL PROGRAM

To ensure accurate laboratory results, strict quality control procedures are enforced. All laboratory quality control data is recorded and reviewed for each analysis. This procedure is specific to the Warzyn laboratory. Any additional quality control requirements for a specific analysis are sited in the Standard Operating Procedure (SOP) manual.

I Quality Control Samples - Internal

A. Selection of Quality Control Samples

1. Spikes and duplicates are to be analyzed on an every 10 or fewer sample basis.

   Example:

1-10 samples	1 duplicate, 1 spike
11-20 samples	2 duplcates, 2 spikes

2. The samples are divided into type of matrix for the selection of quality control. The following criteria are used:

   a. Wet Chemistry and Organics

      Aqueous Samples - Quality control is performed at the 10% frequency (see Section 1).

      Non-aqueous Samples (soils, solid wastes) - The analysis follows the 10% frequency (see Section 1).

   b. Metals

      The difference matrices are analyzed by the appropriate method. The quality control is depended on the matrix. Each matrix must have its own 10% quality control.

      Example:

      Matrices:

      5 Wastewaters 6 Groundwaters 2 Soils

      Quality Control Requirements:

      1 duplicate and 1 spike for the wastewater matrix
      1 duplicate and 1 spike for the groundwater matrix
      1 duplicate and 1 spike for the soil matrix

When the analysis is independent of the matrix (arsenic, selenium, mercury) the 10% frequency is acceptable.

The quality control in furnace work is dependent on the site or project. To identify interferences, each project must have a duplicate and a spike.

3. Low Volume Samples

The low volume sample is noted on the data sheet. If unable to perform the quality control required, an EPA reference sample is carried through the procedure with the samples to document the validity of the method on that day.

4. The Standard Operating Procedure (SOP) manual has the specifications for preparing spikes for each test and further information on quality control for each test.

B. Recording Quality Control Data

1. Quality control charts and tables are based on historical data.

Accuracy - A spike is used to determine the accuracy of the of the analysis. A known concentration of the analyte is added to an aliquot of sample and the percent recovery is calculated as shown below. The control limits for most test procedures are calculated as the average $\pm$ 2 standard deviations. For tests with insufficient data for statistically sound control limits; 85-115% is used for aqueous matrices and 80-120% for non-aqueous matrices. The calculations are performed quarterly or whenever a method is changed.

Spike Calculation - Recovery is calculated as follows:

$$\frac{\text{observed value (spike) - background value (sample)} \times 100}{\text{Known spike concentration}} = \% \text{ Recovery}$$

Precision - A sample is analyzed in duplicate to determine the precision of the analysis. The precision charts and tables are based on the average range between the duplicate values.

Duplicate Calculation - The absolute value of the difference between the 2 values is the range.

observed value 1 - observed 2 = range

The control limits are calculated using the Shewhart UCL calculation.

$$UCL = D_4R$$

$D_4 = 3.27$
R = average value of the range

C. Quality Control Tables

Each test procedure has acceptable quality control ranges which are revised quarterly. It is the analyst's responsibility to check the acceptable ranges after each analysis to ensure the results are in-control.

An out-of-control form is used when a problem occurs during the analysis. It is used to document the process of correcting the problem and to identifiy re-occuring problems. Out-of-control forms are also used for ease of documenting large laboratory projects where all quality control will be reported.

If a quality control data point is outside the acceptable range, the analyst should check for a math error. If a spike/duplicate is still found to be out of control, it should be re-run after the analyst evaluates the test for any obvious interferences. If the re-run sample is still out of control, note it on the data sheet and complete the out-of-control form and notify the laboratory supervisor. A plan of action will be discussed with the quality control officer for resolving the problem.

DOCUMENT ANY UNUSUAL PROBLEMS, SAMPLES, ETC.

See Attached Flow Chart and Out-of-Control form.

II Quality Control Samples - External

A. Warzyn participates in the U.S. EPA Water Pollution and Water Supply blind EPA performance studies each of which occur semi-annually. The results are submitted to the EPA and the laboratory's performance is reported to Warzyn within 2 months of completion of the study.

B. Unknown EPA reference samples are analyzed quarterly. (Results to the analyst.) The samples are analyzed as part of a routine run of samples. This information is recorded and reviewed by the quality control officer.

The reference samples are obtained from:

U.S. EPA Cincinnati, OH 513-684-7325	Environmental Resources Associates 120 East Sauk Trail, Suite 150 South Chicago Heights, IL 60411 312-755-6060

C. Field Quality Control

- One field duplicate is collected for every 10 samples to monitor sampling techniques.
- A minimum of one bailer blank is collected at each site to monitor the cleaning procedures between sampling points.

- For sampling events involving volatile organics, a trip blank and a field blank is also collected.

D. Bottle Quality Control

All samples are collected in new bottles. Most bottles used are pre-cleaned For bottles, which are not pre-cleaned, a quality control check is run each quarter.

E. Quarterly Quality Control Report

A quarterly quality control report is prepared to summarize the laboratory's performance for the last quarter. The report includes a summary of the results of internal and external quality control checks performed for the quarter.

III Standard Curves, Standards, Log Books

A. Standard Curves

Inorganic standard curves consisting of a blank and at least 3 other standards are performed with each analysis. Curves which are calculated using linear regression must have a R2 value of $\geq$ 0.995 and intercept lower than the detection limit.

Organic standard curves consist of a blank and at least 3 additional standards are performed with each analysis. Each standard curve is evaluated separately.

B. Standards

Preparation of standards for each test procedure is specified in the SOP for each method. A check standard is analyzed every 10 samples throughout the analysis. If a check standard is outside of control limits, samples analyzed after the last acceptable check standard are reanalyzed.

C. Log Books

The absorbance or millivolt reading of each check analyzed, the date and the analyt's initials are recorded in the check standard log book. The acceptable ranges are ± 10% for wet chemistry and metals and ± 30% for organic analysis. If a value is outside the acceptable ranges, the lab supervisor must be notified. Log books for daily use, maintenance, temperature, etc. are also kept for various pieces of equipment. Log books are conveniently located throughout the laboratory. The laboratory supervisor is responsible for monitoring this area of quality control.

IV. Data Sheets and Reporting

A. Data Sheets

All data sheets are to be completed the day of the analysis. The sheet is then math checked by an experienced technician who initials the data sheet. The supervisor compares the data to the acceptable limits, initials the sheet and records the quality control data. The data sheet is then ready for final reporting.

B. Reporting

Data is transferred from the raw data sheet to a data summary sheet which is used to summarize all the results of the parameters requested for a particular project. The transferred data is checked by a technician or administrative assistant. As the project is completed, the data summary sheet goes into typing and a final report is created. The final report is checked for errors and if no errors are found, the supervisor approves the report by initializing and dating the report. The report is then issued to the client.

QUALITY CONTROL
CORRECTIVE ACTION FORM

Method:_____

Documentation only____
Reportables effected____

Analyte:_____
Sample Matrix:_____
Lab number(s):_____
_____

	Original	Repeat1	Repeat2, etc.
Batch#:	_____	_____	_____
Date:	_____	_____	_____
Analyst:	_____	_____	_____

Sample set(s) affected (Project#, Lab#):_____
_____
_____

Item(s) affected:	Spike	Duplicate	MS/MSD
Initial Calibration	Linearity	Control Sample	Blank
Endrin/DDT Breakdown	Surrogate	Other_____	
Continuing Calibration			

Problem description:

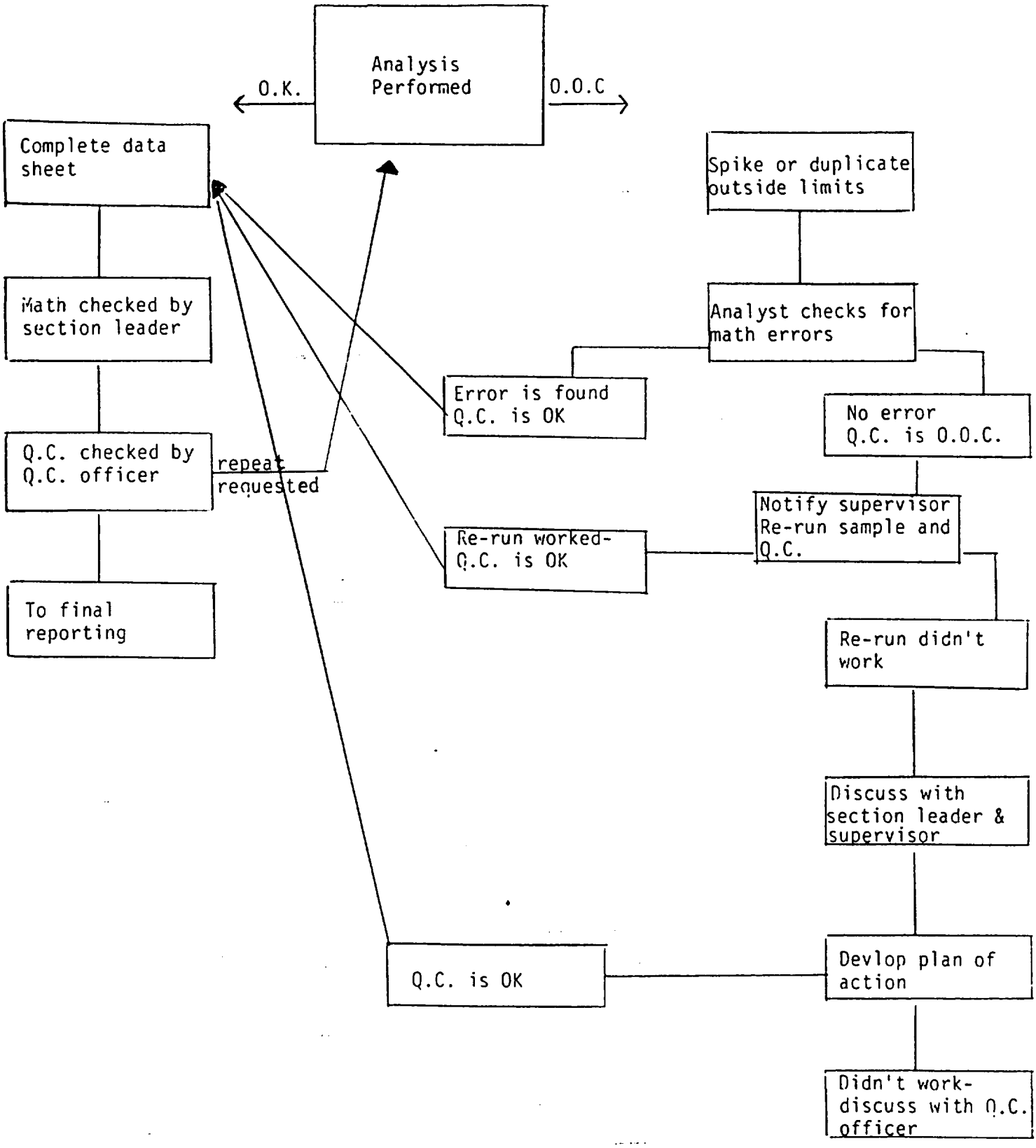
Action taken to solve problem:

Conclusion/effect on reportable data:

Reviewed by technical supervisor:_____ Date:_____
Reviewed by QA officer(when neccessary):_____ Date:_____

QA signature required when data are being reported where associated batch Quality Control is outside of acceptance criteria.

QUALITY CONTROL
FLOW CHART



MAINTENANCE

Daily

Balances (weight check)
pH meters (calibrate)
Refrigerators (temperature check)
D.I. system (conductivity)
Milli-Q system (conductivity)
Conductivity meter

Weekly

Clean balances
Clean hoods
Clean cuvets and flow-through cell (spectrophotometer)

Monthly

Spectrophotometer: Surface cleaning, check absorbance accuracy, check dark current.

Pipettors: Clean plunger and barrel, check for cracks, grease o-ring (replace if necessary), check calibration.

Eye-wash and Shower: Flush both showers and eye-washes, make sure they are in good operating condition.

Dessicators: Surface cleaning, check dessicant and change if necessary.

Ovens and Incubator: Surface cleaning.

Air compressor: Change filters, drain water.

Burets: Soak in soapy water, rinse in pipet washer.

Periodically

Vaccuum pump: Check oil level (quarterly)

Electrodes: Change filling solution (quarterly)

Hoods: Schedule air intake-output check (semi-annually)

Fire extinguishers: Schedule maintenance (annually)

Pipetor - Monthly Maintenance

A. Cleaning

1. Unscrew barrel from pipetor.

2. Inspect glass liner of barrel for cracks (report to section head if found).

3. Clean inside of barrel with alcohol followed by D.I. water. Dry thoroughly, blowing out with air if necessary, to remove any water left in the tip outlet.

4. Check metal plunger for any corrosion. Remove any dirt or corrosion found by gently cleaning with a scrubber. Very lightly, grease the flat surface of plunger.

5. Inspect O-ring for signs of wear (cracking, discoloration, etc.) and replace if necessary (see manufacturer's instructions).

6. Grease O-ring very lightly, with the lubricant provided and reassemble pipetor.

B. Calibrating

1. After the monthly maintenance has been performed, dispense five 2 mL volumes of water into a dry 10 mL graduated cylinder. Repeat procedure with a second cylinder. The volumes should read 10.0 mL $\pm$ 0.5 mL.

2. Dispense five 5 mL volumes of water into a dry 25.0 mL graduated cylinder. Repeat procedure with a second cylinder. The volumes should read $\pm$ 1.0 mL.

3. Record volumes in maintenance log. If the volumes obtained are not within the specified ranges, check the volume set on pipetor and readjust if necessary, and repeat the calibration check.

   If the volume accuracy is still not correct, repeat the maintenance procedure and change the O-ring if that has not already been done. Repeat calibration procedure.

Note: At any time during the month, if the pipetor feels sticky or sluggish, or if any liquid is drawn up into pipetor, maintenance should be performed immediately.

Revision Dates

Michael Linskens 9/16/87
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Laboratory Manager

Kim Finner
Kim D. Finner
Analytical Laboratory QA/QC Officer

Lawrence D. Andersen
Vice President, Technical Services

PIPETOR MAINTENANCE & CALIBRATION

	Maintenance Performed																				
W.C. A	Vol: 2 mL 5x																				
	Vol: 5 mL 5x																				
	Maintenance Performed																				
W.C. B	Vol: 2 mL 5x																				
	Vol: 5 mL 5x																				
	Maintenance Performed																				
Metals A	Vol: 2 mL 5x																				
	Vol: 5 mL 5x																				
	Maintenance Performed																				
Metals B	Vol: 2 mL 5x																				
	Vol: 5 mL 5x																				

LAB MAINTENANCE - 6 MONTH SCHEDULE

Maintenance Description	August	September	October	November	December	January
Balances: Surface Cleaning						
Hoods: Surface Cleaning						
Spectrophotometer: Clean cuvets and flow-through cell						
Air Compressor: Change filters Drain water						
Burets: Wash and D.I. rinse						
Dessicators: Surface Cleaning Change Dessicant						
Eye Wash & Shower: Flush thoroughly						
Ovens and Incubator: Surface Cleaning						
Pipettors: Clean and check Calibration						
Spectrophotometer: Surface Cleaning, Abs. check, dark current check						
Electrodes: Change filling solution		X	X		X	X
Fire Extinguisher: Schedule maintenance		X	X	X	X	X
Hoods: Schedule flow rate check		X	X	X	X	X
Vaccuum Pump: Check oil level		X	X		X	X

LACHAT AUTO-ANALYZER MAINTENANCE

Week of:*												
Tubing: Checked and changed if necessary (daily)	M	T	M	T	M	T	M	T	M	T	M	T
	W	Th	W	Th	W	Th	W	Th	W	Th	W	Th
	F	S	F	S	F	S	F	S	F	S	F	S
Waste container: Empty & rinse for new manifold (daily)	M	T	M	T	M	T	M	T	M	T	M	T
	W	Th	W	Th	W	Th	W	Th	W	Th	W	Th
	F	S	F	S	F	S	F	S	F	S	F	S
Manifolds: Rinsed with D.I. water after use (daily)	M	T	M	T	M	T	M	T	M	T	M	T
	W	Th	W	Th	W	Th	W	Th	W	Th	W	Th
	F	S	F	S	F	S	F	S	F	S	F	S
Pump: Sprayed with silicon (weekly)												
Data file: Back-ups made (weekly)												
Machine area: Cleaning (weekly)												

* Give Monday's date of each week and initial each day maintenance is performed.

INORGANIC - DAILY QUALITY CONTROL PROCEDURE

Scope and Application: Daily check of desiccant, instruments, refrigerators and freezer, deionized and Milli-Q water systems to ensure data produced each day is of top quality.

Desiccant: Dri-Rite, an anhydrous compound, is kept in the bottom of each desiccator. When Dri-Rite is blue, it is ready to use, but when a pink shade begins to form, it must be dried in an oven set at 103-105°C until no pink color can be detected. Record date dried in comments section. Record initials and date of daily check in the log book.

Refrigerators and Freezer: Temperature of each of the refrigerators are to be recorded in the equipment record book daily.

Instrument	Acceptable Range
Walk-in cooler #1	4.0°C ± 2.0°C
Walk-in cooler #5	4.0°C ± 2.0°C
Silver Raetone #2	4.0°C ± 2.0°C
Gold General Electric #3	4.0°C ± 2.0°C
White Frigidaire #4	4.0°C ± 2.0°C
BOD Incubator	20.0°C ± 1.0°C

Notes:

1. Each of the refrigerators, freezers and incubators should routinely be defrosted when ice build-up occurs.

2. If fluctuations in temperature do occur, report the occurrence to the lab supervisor immediately. Note in maintenance records.

Instruments: In-lab balances, pH meter, and conductivity meter are to be checked daily against known standards. An Instrument Operating Procedure (IOP) is located in each of the instruments representative log book.

Balance: The Sartorius and Fisher balances are calibrated with Class "S" weights. The weights are to be handled with plastic forceps. Each weight is to be recorded in its representative place in the log book. Acceptable ranges are listed in each IOP.

pH Meter: The pH meters are calibrated with fresh buffers daily according to the pH SOP. Record the reading of the pH 10.00 buffer in the log book also. The acceptable range for the pH 10.00 buffer is 9.90 - 10.10.

Note: The inner electrode solution should be checked periodically to ensure accurate readings.

Conductivity Meter: The Lab-Line or the YSI conductivity meter is checked daily against a KCL standard of known concentration. Check the meter according to the IOP located in the log book. The acceptable range is 90-110% of the true value.

Deionized and Milli-Q Water Systems:

D.I.: The D.I. system functions with ion-exchange cartridges located in the sample storage room. Two warning lights are attached to the tanks to ensure they are operating properly. Each day, these lights must be checked. If one or both are out, the supplier must be notified to replace the tanks. Record tank change dates in the log book.

Note: If both lights are out, D.I. water cannot be used until tanks are replaced.

Milli-Q: The Milli-Q water should be checked with a conductivity meter. Conductivity should be no greater than 1 umhos/cm @25°C. The Milli-Q system calibration must also be checked daily. This is done by turning on system and letting pressure build, then depress test button. Reading should be at least 10 megohms/cm. Record in log book. (16 megohms/cm maximum load).

Revision Date

8-18-87

Kim Johnson — 5-22-91

Kim D. Johnson
Analytical Laboratory Manager

Michael Linskens

Michael J. Linskens
Director, Analytical Services

Daily Desiccant Quality Control Check

Date	Initials	Desiccator ID	Desiccant Blue	Desiccant Pink	Comments

Effective Date: 1-14-92

CHAIN-OF-CUSTODY
Superfund Level

Scope and Application: Chain-of-custody procedures are used to maintain and document sample possession. This particular procedure is applicable when full chain-of-custody procedures are required for enforcement driven investigations.

Reference: **NEIC Policies and Procedures, May, 1978 (Revised March, 1986).**
EPA-330/9-78-001-R.
U.S. EPA Central Regional Laboratory (10/15/82).

Notes: Due to the legal nature of enforcement driven investigations, possession of samples must be traceable from the time the samples are collected until introduced as evidence in legal proceedings or destroyed. To maintain and document sample possession, strict chain-of-custody procedures are followed.

A sample is under your custody if:

1. It is in your possession, or
2. It is in your view, after being in your possession, or
3. It was in your possession and you locked it up, or
4. It is in a designated secure area.

Field Custody:

1. As few people as possible should handle samples.

2. The field sampler is personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched to the appropriate laboratory.

3. Sample tags and labels shall be completed for each sample, using water proof ink (unless prohibited by weather conditions). **For example:** a log book notation would explain that a pencil was used to fill out the sample tag/label because a ball point pen would not function in freezing weather.

4. The field project coordinator determines whether proper custody procedures were followed during the field work and decides if additional samples are required.

Transfer of Custody and Shipment:

1. Samples will be packaged properly for shipment and dispatched to the appropriate laboratory for analysis, with a separate custody record accompanying each shipment. The method of shipment, courier name, and other pertinent information is entered in the "Remarks" (#14) section of the custody record (see Figure 3). Shipping containers are "sealed" with numbered chain-of-custody seals. The seal numbers are also noted in the "Remarks" section of the custody record.

2. Samples are accompanied by a chain-of-custody record (see Figure 3). When transferring the possession of samples, field personnel will complete the information required for #1 through #11 on the chain-of-custody. Any remarks pertaining to shipping are included in #14. This information is required prior to the field personnel relinquishing custody of the samples. This record documents sample custody transfer from the sampler, to the mobile laboratory or a distant laboratory.

3. Each shipping container will be accompanied by a chain-of-custody record identifying the contents. The original record will accompany the shipment, and a copy will be retained by the field project coordinator.

Sample Receipt and Sample Log-In Procedures:

Safety Precautions: All samples received should be considered hazardous and appropriate precautions should be taken when handling these samples. Under no circumstances should any personnel other than the sample custodian or project leader open coolers. If damage or leakage is noted, stay clear of the coolers and notify the sample custodian or project leader immediately. All samples will be opened in a hooded area!

Sample Receipt:

1. The project leader will notify the sample custodian in writing of incoming samples.

2. The custodian will receive the samples and deliver them to a hood located in the analytical laboratory. The procedures described below will be followed by the sample custodian:

   a. Examine the shipping container and record the following information on the project log form (Refer to Figure 1). A separate project log form is required for each shipping container.

      - The presence/absence of custody seal on the shipping container.

      - The condition of the custody seal (i.e., intact, not intact).

   b. Open the shipping container in a hooded area, remove the enclosed sample documents, and record the following information on the project log form.

      - The presence/absence of the chain-of-custody record(s).

      - The presence/absence of airbills and/or bills of lading documenting shipment of the samples.

   c. Remove the samples from the container and record the following information in the project log form.

      - Condition of samples (intact, broken, leaking, cold, etc).

      - The presence/absence of sample tags.

- Sample tag numbers. Compare these numbers with the chain-of-custody record(s) (Figure 3). If sample tag numbers do not match. Record this fact on both the chain-of-custody record and project log form and contact the operations manager so that the discrepancy can be resolved.

d. Compare the following documents to verify agreement of the information contained on them.

- Chain-of-custody records.
- Sample tags.
- Airbills or bills of lading.

e. Sign the Chain-of-custody (Figure 3 - #12 and #13), project log form and airbills.

Log-In and Tracking:

Note: The laboratory is a secured area with strict limited access. Any samples arriving to the laboratory during "off hours" are stored in the laboratory refrigerator until they are logged in. Laboratory personnel are responsible for the care and custody of the samples during the analysis stage and until the samples are removed from the refrigerator and properly disposed of.

1. **Log-In Procedure:** Samples are logged in as follows (Refer to Appendix 1):

   a. **Sample Information:** Care should be taken to document the condition of samples upon receipt. Anomalies such as broken or leaking bottles, expired holding times, improper labeling or preservation, and air bubbles in VOA vials should be noted on the chain-of-custody. It should also be noted whether the samples arrived cold. The project manager will be informed about discrepancies and will determine action to be taken.

   b. **Numbering Samples:** Number the samples on the chain-of-custody form. Record lab numbers used in the black lab book.

Note:
- Sample preparation is required if the samples need to be filtered and/or preserved in the lab.
- If metals are requested, metal digestion is required for all matrices.
- Sample compositing is required if the lab is instructed to composite a series of samples received into one sample for analysis.

   c. **Subcontracted Work Required:** Refer to the subcontracting SOP for further instructions on subcontracting samples.

   d. **Special Accounting Notes:** If there is a discount or a special proposal for the sample analyses to be charged, record that information on the chain-of-custody.

e. **Labeling Bottles:**

- Record the lab number on the sample bottle label, tags and caps using a water-proof marker.
- Check that the appropriate preservation and preparation has been circled.
- If metals and total hardness are both required, using a red water-proof marker, place a dot on the bottle cap of that bottle to be used for those analyses.

f. **Preserving Samples:** Samples which have been preserved in the field must be checked via pH paper in the laboratory to insure they were preserved to the correct pH. Refer to project QAPP for preservation and bottle requirements. The project manager will be notified about any incorrectly preserved samples. Check preserved samples as follows:

- Acid-preserved (HNO_3 or H_2SO_4) samples must be preserved to a pH <2. Using a pH stick, check the pH. If the pH is not <2, note this on the chain-of-custody, then add additional acid until a pH is obtained. **It is critical that the correct type of acid be added.**
- Base-preserved NaOH samples must be preserved to a pH $>$ 12. Using a pH stick, check the pH. If the pH is not $>$ 12, note this on the chain-of-custody, then add additional 10N NaOH until a pH $>$ 12 is obtained.
- Base-preserved NaOH with ZnAc samples must be preserved to a pH $>$ 9. Using a pH stick, check the pH. If the pH is not $>$ 9, note this on the chain-of-custody, then add additional 10N NaOH until a pH $>$ 9 is obtained.

1. Samples which have not been preserved completely in the field need to be preserved in the laboratory. Record any preservation done in the lab on the field sheets or custody records. It is important that samples arrive in appropriate containers (refer to project QAPP). If they do not, contact the inorganic/organic supervisor. They will determine which analyses, if any, can be run.

Computer Log-In:

1. Analysis requests are entered into the Laboratory Information Management System (LIMS). The LIMS database is used for scheduling of analytical work and to produce accounting invoices. Analysis requests are entered into the LIMS database as follows:

Computer Log-on

- Turn on the computer terminal.
- Enter user name > last name [return].

- Local prompt > c_chico [return].
- User name: ACSORA [return].
- Password: [return].
- Please enter your last (15 characters max.): Last name: [return].
- $Labmenu [return].
- Username: Last name [return].
- Password: [return].
- Select Log-in/Order entry and press [commit].

2. The log in screen will appear as shown in figure 2. Using information on the chain-of-custody, proceeds as follows:

   a. Enter project number (return). Account number and name will be filled in automatically. If the information matches the project name on the chain-of-custody, press return.

   b. Sample date automatically defaults to yesterday's date. If this is correct, press return or override with correct date.

   c. Enter sample identification from **station location** on the chain-of-custody and press return.

   d. Enter proper matrix number and press return.

   e. **Desc:** This is used if sample identification is longer than the number of characters in the sample identification field. (Return to override or enter information and return.)

   f. Date received automatically defaults to today's date. If correct, press return or override with correct date.

   g. Days to process automatically defaults to 14 days. If this is correct press return or for standard turnaround time (usually 4 weeks) or if rush work, (indicated on request form) override with the number of days until due.

   h. **Duedate:** First date listed is report date, second date is the lab due date which can be changed if necessary by changing days to process.

   i. **Comments:** Enter "! CLP Protocol", special tests or other information needed to analyze samples correctly and press return.

   j. **Manager:** Filled in automatically; press return if correct or override.

k. **Markup/Discount:** Return if correct or override.

- % Markup/Discount: For accounting. Enter a negative number for a discount (i.e. -15 for 15%) and a positive number for markups (i.e. 100 for 2 x price). Check accounting sheets for projects with special pricing.

l. To enter parameters, press **Next Screen** (see Figure 2A).

Note: To move from block to block press Next/Previous screen. To move from field to field, press Return/Tab or F12 (previous field) key.

m. Matrix automatically defaults to matrix entered above (return or override).

n. Enter the proper code name for the test to be run from report MF01. Use parent codes if applicable for quicker entry. (Press the down arrow key, return.) To delete use remove key.

o. To enter address for the report, press **Next Screen** (see Figure 2B).

p. **Type:** Report (return).

q. If an address has already been entered and matches the chain-of-custody press **Commit** and return.

r. If no address appears, press return until cursor is in the City field. Type in the city and press return.

s. Type in state (press **Commit** and return). After city and state have been entered for the first sample, it will automatically be copied for the rest of the sample numbers in a single log-in.

3. To duplicate the entry for another sample on the same chain-of-custody:

a. If next sample to be entered has all the same information as the previous sample entered, press **Duplicate Record** key (F7).

b. Enter sample identification and press **Commit** key, return.

4. To duplicate only the information in the upper block of the log-in screen (sample date, matrix etc.):

a. Press **Create Record** key (Insert Here) and enter sample identification.

b. Press **Previous Screen** (F12) if sample date needs to be changed.

c. Press **Next Screen**, return. Then press **Next Screen** again to enter new product codes for the analyses requested.

d. Press **Previous Screen** to enter new sample or to quit.

e. Press **Clear Form** (F17) to enter a new chain-of-custody (new log in number).

f. Press **Exit/Cancel** (PF4) to exit from the log-in screen.

- Press **Exit/Cancel** to exit from the LIMS system.
- At the VMS $ prompt, enter LO (return) to log off the VAX computer.
- Enter N (return).

5. To print daily log-in reports:

   a. Print out the appropriate FS02 reports for only the samples logged in that day. The computer printout must be checked against the custody record or field sheets by the inorganic/organic supervisors so that any errors can be corrected. Print a report as follows:

   b. Select "Seedpak Reporting" from the main menu, and press **Commit**.

   c. Select FS02 and press **Commit**.

   d. Enter today's date, press **Commit** and return.

   e. The inorganic/organic supervisors are responsible for updating and printing lab schedules as they require them.

   f. Any "rush" work is to be brought to the inorganic/organic supervisors' attention immediately.

Sample Storage:

1. Samples and extracts will be stored in a secure area designated for strict chain-of-custody samples.

2. Damaged samples will be disposed of in an appropriate manner and the method of disposal documented.

3. The laboratory and sample storage areas are secure with strict limited access by only laboratory personnel.

4. Whenever samples are removed from storage, the removal will be documented. All transfers of samples will be documented on the internal chain-of-custody records. (Refer to Figure 4).

5. Samples and extracts will be stored after completion of analysis in accordance with the contract or until sample custodian is instructed otherwise by the enforcement Project Officer.

6. The location of stored organic extracts will be recorded.

7. VOA samples will be stored separately from organic extracts.

8. Standards are stored separate from samples.

9. Samples requiring refrigeration are stored in the walk-in coolers refrigerator. Metal samples are stored in the black cabinets in log-in. Metal digestates are stored on shelves in the metals lab.

Sample Security:

1. Samples will be stored in a secure area.

2. Access to the laboratory will be through a monitored area. Other outside-access doors to the laboratory will be kept locked.

3. Visitors will sign a visitors log (located at the reception area and will be escorted while in the laboratory area.

4. Refrigerators, freezers, and other sample storage areas are secure as they are located within the laboratory.

5. Only laboratory personnel have access to the laboratory.

6. Samples will remain in secure sample storage until removed for sample preparation or analysis. All transfers of samples into and out of storage will be documented on an internal chain-of-custody record. An example of an internal chain-of-custody record used for this procedure is attached. (See Figure 4).

7. These internal custody records will be maintained in the project file.

8. After a sample has been requested from storage by the analyst, the analyst is responsible for the custody of the samples. Each analyst must return the samples to the storage area before the end of the working day.

Internal Chain-of-Custody: The following procedure for documentation of internal chain-of-custody for samples requiring continuous custody.

1. All transfer of samples into or out of the storage areas will be documented on an internal chain-of-custody record (see Figure 4). These records are maintained by the sample custodian.

2. Once a sample is removed from a walk-in by the sample custodian, he/she will then give it to the analyst, who is then responsible for the custody of the sample. Each analyst **must return** samples to the walk-in **before the end of the working day**. Samples are not allowed to sit on the bench overnight.

3. The next morning, the sample custodian will put samples back in their proper place on the shelves. Sample custodian is responsible for signing sample request sheet that samples were returned properly. Any remarks will also be noted by the sample custodian.

4. When sample analyses and necessary quality assurance checks have been completed by the laboratory the unused portion of the sample should be disposed of properly. (See sample disposal section.) All identifying tags, data sheets, and laboratory records shall be retained as part of the permanent documentation of the project. **Tags and forms are never disgarded!**

Sample Disposal:

1. Due to limited sample storage facilities available, the following guidelines have been created for sample storage/disposal. Sample storage time may be extended upon client's request.

   a. Two months after the report issue date, aqueous inorganic samples which are in cold storage are to be transferred to warm storage.

   b. Two months after the report issue date, aqueous organic samples which are in cold storage are to be transferred to warm storage.

   c. Three months after report issue date, soil samples which are in cold storage are to be transferred to warm storage.

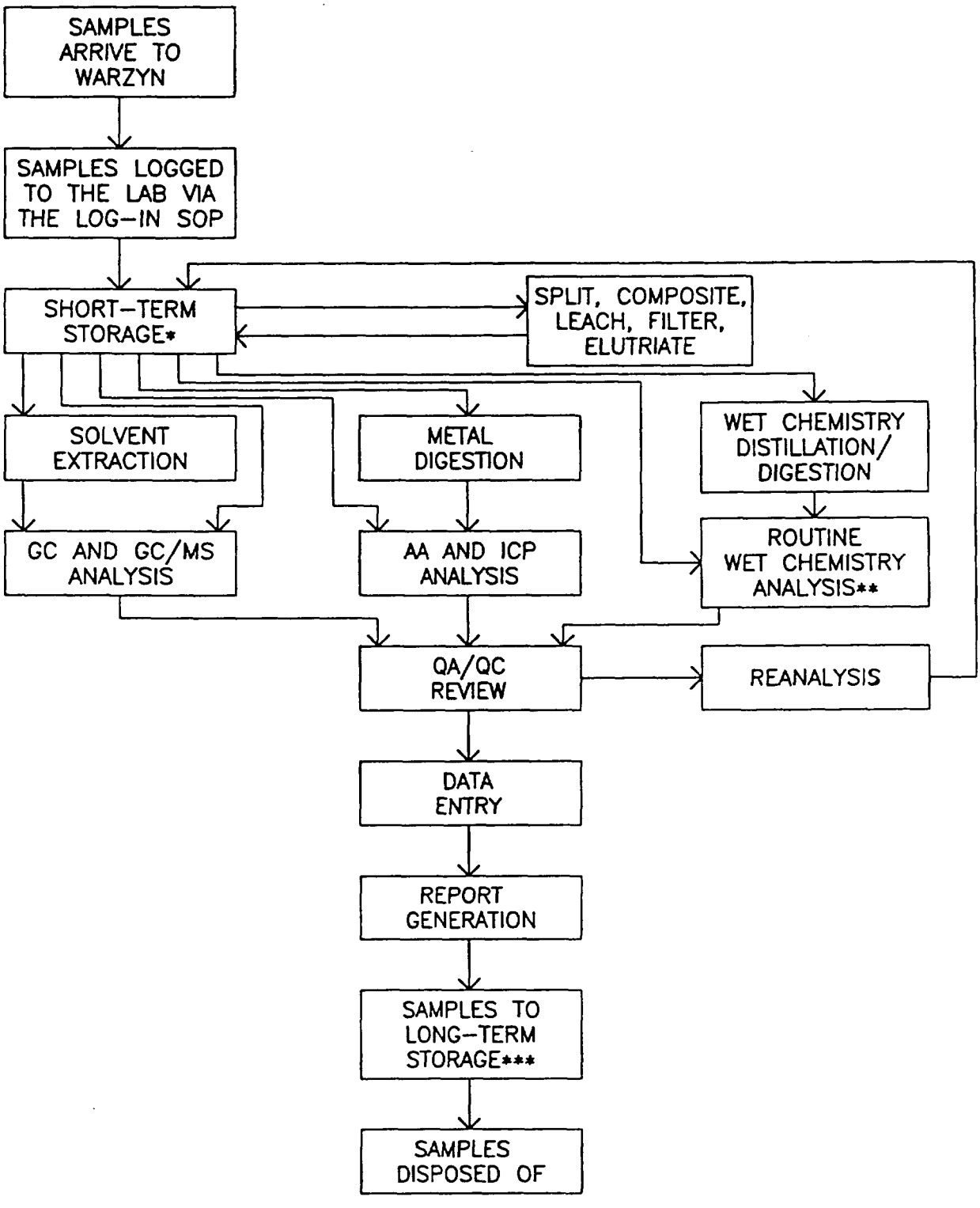
   d. Samples in warm storage should be disposed of as follows:

      - Aqueous Samples: Two months after report issue date, a memo is sent to the project manager stating that the samples will be disposed of 1 month from the date of the memo. The samples are then disposed of 3 months after report issue date unless project manager requests alternate arrangements.

      - Non-Aqueous Samples: Six months after report issue date, a memo is sent to the project manager requesting permission to dispose of the samples. This memo is repeated on a quarterly basis until project manager approves sample disposal.

Warzyn Inc.
Analytical Services
Sample Flow Diagram

Appendix 1



* SHORT TERM STORAGE: WALK-IN REFRIGERATOR FOR SAMPLES REQUIRING REFRIGERATION, BLACK CABINETS IN SAMPLE LOG-IN AREA FOR SAMPLES NOT REQUIRING REFRIGERATION, WALK-INS FOR SAMPLES REQUIRING "SUPERFUND LEVEL CHAIN-OF-CUSTODY".

** ROUTINE WET CHEMISTRY ANALYSES INCLUDE BOTH AUTOMATED AND MANUAL TESTS

*** LONG TERM STORAGE: NON-REFRIGERATED STORAGE IS LOCATED IN THE FIELD STORAGE ROOM.

WARZYN

FIGURE 1

Sample Custodian Signature: ____________________
Date: ____________________

(Circle Appropriate Response)

Custody Seal — present/absent
intact/not intact

Chain-of-Custody — present/absent

Sample Tags — present/absent
Sample Tag Numbers — listed/not listed on chain-of-custody

Project Number: ____________________

Airbill Number: ____________________

Date Rec'd	Time Rec'd	Chain-of Custody Record No.	Corresponding		pH <2	pH >9	pH >12	Does info. on Custody Records, Traffic and Sample Tags Agree	Remarks Condition of Samples
			Sample Tag Nos.	Assigned Nos.					

FIGURE 2

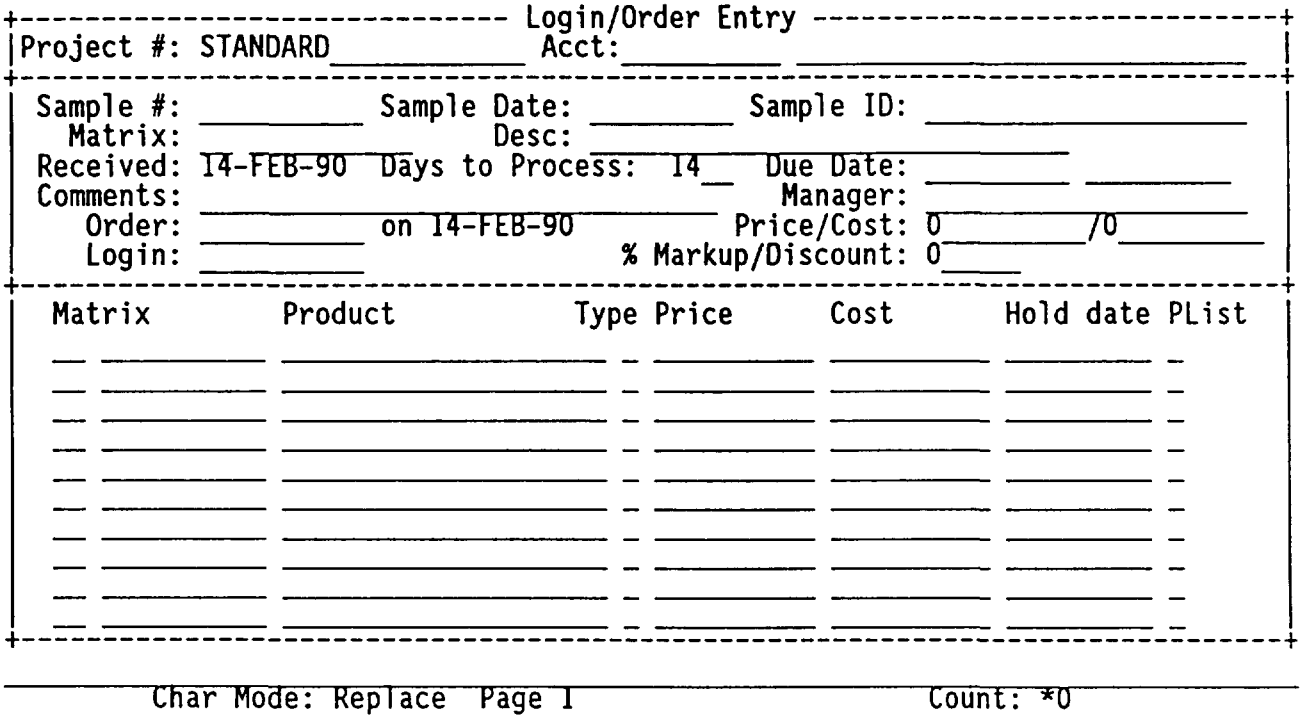

Login/Order Entry
Project #: STANDARD
Acct:
Sample #:
Sample Date:
Sample ID:
Matrix:
Desc:
Received: 14-FEB-90
Days to Process: 14
Due Date:
Comments:
Manager:
Order:
on 14-FEB-90
Price/Cost: 0/0
Login:
% Markup/Discount: 0
Matrix
Product
Type
Price
Cost
Hold date
PList
Char Mode: Replace
Page 1
Count: *0

FIGURE 2A

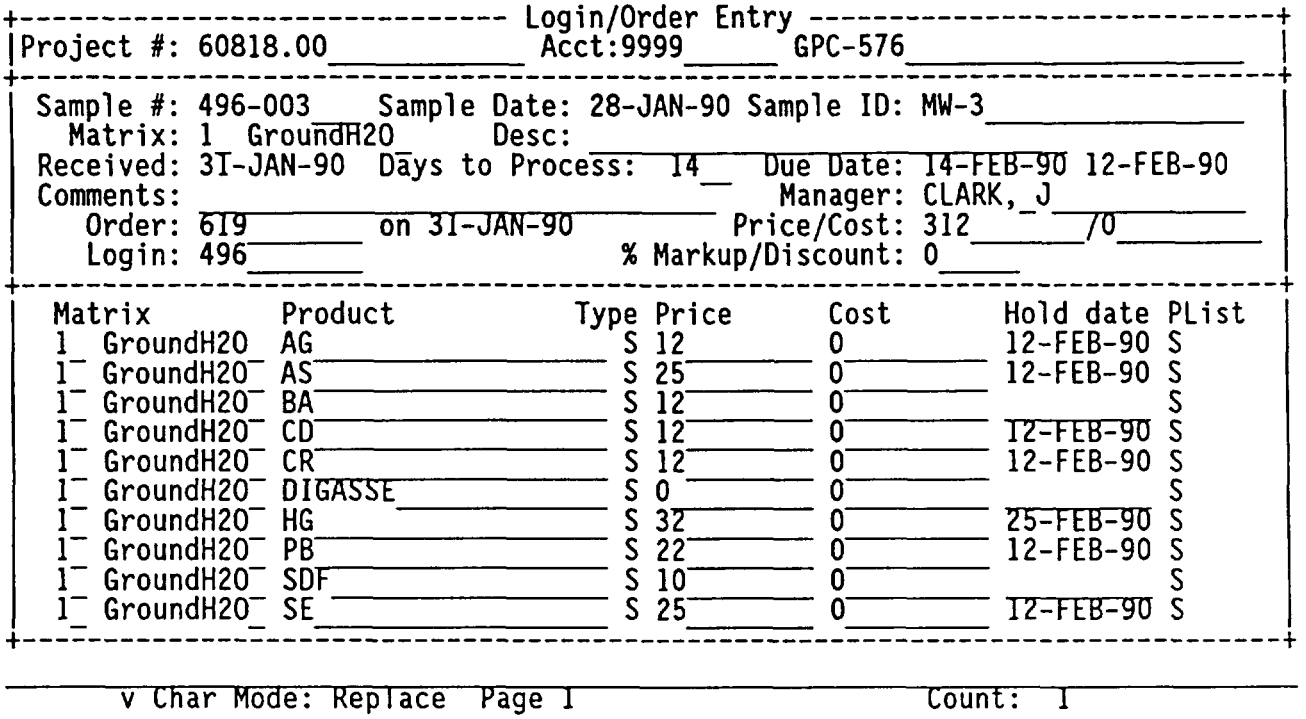
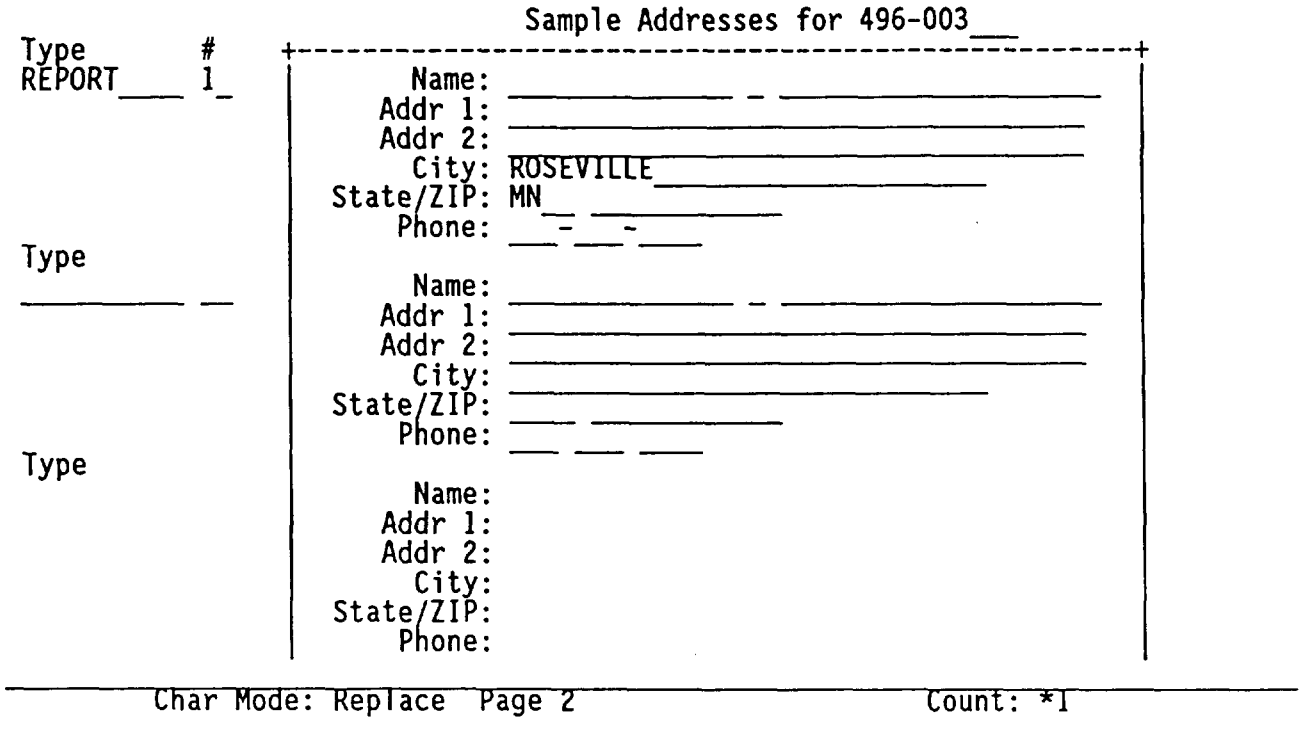

Login/Order Entry
Project #: 60818.00 Acct:9999 GPC-576
Sample #: 496-003 Sample Date: 28-JAN-90 Sample ID: MW-3
Matrix: 1 GroundH2O Desc:
Received: 31-JAN-90 Days to Process: 14 Due Date: 14-FEB-90 12-FEB-90
Comments: Manager: CLARK, J
Order: 619 on 31-JAN-90 Price/Cost: 312 /0
Login: 496 % Markup/Discount: 0
Matrix Product Type Price Cost Hold date PList
1 GroundH2O AG S 12 0 12-FEB-90 S
1 GroundH2O AS S 25 0 12-FEB-90 S
1 GroundH2O BA S 12 0 S
1 GroundH2O CD S 12 0 12-FEB-90 S
1 GroundH2O CR S 12 0 12-FEB-90 S
1 GroundH2O DIGASSE S 0 0 S
1 GroundH2O HG S 32 0 25-FEB-90 S
1 GroundH2O PB S 22 0 12-FEB-90 S
1 GroundH2O SDF S 10 0 S
1 GroundH2O SE S 25 0 12-FEB-90 S
v Char Mode: Replace Page 1 Count: 1

FIGURE 2B


Sample Addresses for 496-003
Type
REPORT
#
1
Name:
Addr 1:
Addr 2:
City: ROSEVILLE
State/ZIP: MN
Phone:
Type
Name:
Addr 1:
Addr 2:
City:
State/ZIP:
Phone:
Type
Name:
Addr 1:
Addr 2:
City:
State/ZIP:
Phone:
Char Mode: Replace Page 2
Count: *1

WARZYN

FIGURE 3

CHAIN OF CUSTODY RECORD

Warzyn Engineering Inc.
One Science Court
University Research Park
P.O. Box 5385
Madison, Wisconsin 53705
(608) 273-0440

PROJ. NO.	PROJECT NAME # 2A	NO. OF CONTAINERS #8							REMARKS
#1	LOCATION: # 2B								
SAMPLERS: (Signature) #3									
LAB NO. #15	DATE #4	TIME #5	COMP.	#6 GRAB	#7 STATION LOCATION				#9

Relinquished by: (Signature) #10	Date / Time #11A / #11B	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature) #12		Date / Time #13A / #13B	

Remarks #14

PROJECT MANAGER: #15

Figure 4

WARZYN SAMPLE REQUEST SHEET

Requested By: _____ Need Samples By:
Date/Time Submitted: _____ Date: _____
Time: _____

I am requesting:

Project No.	Sample No(s)	Parameter	Matrix	Remarks

I am returning: Date/Time:

Project No.	Sample No(s)	Remarks

APPENDIX F

GENERAL STANDARD OPERATING PROCEDURES FOR HAUSER LABORATORY, INC.

MEMORANDUM OF CALL

Previous editions usable

TO: KEVIN B.

☐ YOU WERE CALLED BY– ☒ YOU WERE VISITED BY–

NAN GOWDA

OF (Organization)

☒ PLEASE PHONE ▶ ☐ FTS ☐ AUTOVON

☐ WILL CALL AGAIN ☐ IS WAITING TO SEE YOU

☐ RETURNED YOUR CALL ☐ WISHES AN APPOINTMENT

MESSAGE

I received the attached SOPs for the LENZ OIL SITE. DAVID EDWARDS OF ERM WANTS TO HAVE A CONF. CALL WITH YOU. PLEASE ~~CALL~~ GIVE ME A CALL ASAP THANK! Nan

RECEIVED BY	DATE	TIME
	1/16/91	1:30

63-110 NSN 7540-00-634-4018 **STANDARD FORM 63 (Rev. 8-81)**
Prescribed by GSA
FPMR (41 CFR) 101—11.6

* U.S. GPO 1990 — 262-080

ERM - NORTH CENTRAL, INC.

Letter of Transmittal

102 WILMOT ROAD, SUITE 300 DEERFIELD, ILLINOIS 60015 (708)940-7200

TECHNICAL SUPPORT SECTION
DOCUMENT

TO Mr. Nat Gauda
Project Manager
USEPA - Region V, HSRL-6J
77 W. Jackson
Chicago, Illinois 60604

DATE	PROJ. NO.
1-15-92	0252DE
ATTENTION	
RE: Lenz Oil RI/FS	
QAPP Addendum - Revision 1A	

WE ARE SENDING YOU

☒ Attached ☐ Under separate cover via _____ the following items:

☐ Shop drawings ☐ Technical Paper ☐ Prints ☐ Plans

☐ Copy of letter ☐ Samples ☐ Specifications ☐ _____

COPIES	DATE	NO.	DESCRIPTION
1			CompuChem's SOP for Chain-of-Custody Procedures
1			Warzyn's SOPs for Analyzing Residential well samples for
			low-concentration inorganics.

THESE ARE TRANSMITTED AS CHECKED BELOW:

☐ For approval ☐ Approved as submitted ☐ Resubmit _____ copies for approval

☐ For your use ☐ Approved as noted ☐ Submit _____ copies for distribution

☐ As requested ☐ Returned for corrections ☐ Return _____ corrected prints

☒ For review and comment ☐ _____

REMARKS The Chain-of-Custody SOP for CompuChem is enclosed for QAS's review prior to resubmittal of the QAPP Addendum. Warzyn informed us that they perform analyses of residential well samples for low-concentration inorganics using the enclosed SOPs rather than ILC 01.0 as stated in the QAPP Addendum. Please pass these on to Kevin Bolger for his review and comment. We can discuss them tomorrow.

COPY TO _____ SIGNED Dan P. Eklund

If enclosures are not as noted, please notify us immediately

CompuChem's

Chain-of-Custody Procedure

Appendix D

Production Planning & Control SOP 1.1: Logging In Samples

The following steps are completed for all samples as they are received by CompuChem Laboratories. If for any reason a sample requires special handling upon receipt, the Manager of Production Planning and Control is consulted for directions as to the proper handling and documentation of the samples.

- Before opening and while inspecting each sample, each employee is required to wear protective clothing (lab coat, safety glasses and gloves). These items need to be worn at all times when in the marked areas (colored line).

- Each sample container is inspected before opening, making sure that it has not been damaged or opened during shipment. For those clients using padlocks, sealing tape, or custody seals, these items are inspected to make sure that they are intact and this observation is recorded on the chain-of-custody form (Attachment 1). If the custody seals, tapes, or padlocks are broken, one must contact Customer Service (for commercial samples) or the Sample Management Office (for EPA samples) for permission to continue processing the sample.

- Each container is opened under the hood and checked for breakage. The condition of the refrigerant is checked (whether any ice remains or whether the cooling packs are solid), and the temperature is obtained by adhering a calibrated temperature strip to the outside of one representative sample container within each cooler. The temperature is taken and recorded on the Sample Record (Attachment 2) per Production Planning and Control SOP 3.8; Reading and Recording Temperature of Samples.

- The temperature and pH are recorded on the Log-In Sheet. If a temperature or pH variance occurs, a QA Notice is written and associated with the sample (Attachments 3-4).

- Receiving personnel must sign and date all chain-of-custody documentation upon sample receipt and record any discrepancies (sample matrix, for instance) on the chain-of-custody form.

- The Supervisor of Sample Receiving must verify that the Receiving Clerk has signed and dated the chain-of-custody form.

- When a CompuChem SampleSaver is received, the SampleSaver number is recorded on the Log-In Sheet and is entered into the LMS system by number.

- Samples are removed from the shipping container and the sample identification information on the sample bottles is compared to the sample information on the traffic sheets, packing lists, and Chain-of-Custody Form included in the container (Attachments 5-11).* For EPA samples, Form DC-1 is filled out as well, per USEPA SOW. If discrepancies exist, the problem is noted on the Chain-of-Custody Form; the Receiving Clerk notifies Customer Service (for commercial samples) and the Supervisor notifies SMO (for EPA samples).

- Each water VOA is checked for air bubbles and headspace, and noted on the Chain-of-Custody Form as well as the Log-In-Sheet.

- On each complete and correct EPA Chain-of-Custody and Traffic Report the statement "Received in Good Condition" is written or stamped, initialed and dated by the receiving individual.

- On each complete and correct Commercial Chain-of-Custody the statement "Received in Good Condition" is written or stamped, initialed and dated by the receiving individual. "Received in Good Condition" is intended to indicate that the sample or samples were received intact with all associated sample tags (if applicable), custody seals (if applicable), pH for inorganics, and corresponding documentation in order. If there are any discrepancies in the documentation or other problems (such as breakage of the containers or chain-of-custody seals), the exceptions are noted on the appropriate documents, initialed and dated.

- The statement "Received in Good Condition" does not, however, include sample temperature since EPA samples are generally received at temperatures above the recommended 4°C. The temperature is noted on the sample Log-In Sheets and the gray envelope.

- Incoming samples are checked against SMO scheduled receipts (for EPA samples).

* NOTE: For samples associated with NJDEP X-408, an NJDEP059 "Sample Analysis Request Form" will accompany the samples upon receipt (Attachment 12).

- The sample is logged in on the Log-In Sheet Log, noting the following items:

Case number	Temperature
CompuChem sample ID	Client name or order number
Receiving date (RD)	Sampling date (SD)
Analysis codes	Matrix
Volume received	pH (Inorganics Samples Only, see PP&C SOP 3.1)

- For EPA samples, the samples' account data is entered into the marketing section of the CLMS in order to generate the order number and associated requisition numbers. For commercial samples, customer service is contacted to check for the existence of the order and to receive requisitions for analyses. The order is then completed in the CLMS, and the EPA Scheduling Log (Attachment 13) is also completed.

- The sample is entered into the sample receipt portion of the CLMS in order to generate a CompuChem number for each sample. The CompuChem number is filled in on the accessioning log sheet (this completes the log sheet).

- The CompuChem number is a unique, six-digit identity that is generated by the LMS system in numerical sequence. It can be cross-referenced to the Client ID.

- A CompuChem label is generated in numerical sequence, with the CompuChem number.

- The samples are labelled with the CompuChem number by wrapping each sample bottle with its computer-generated CompuChem sample label. Sample labels are color-coded, and are to be rotated with a different color every two-week period by the Supervisor of Receiving or the Supervisor's designee.

- Each Log-In Sheet is reviewed by the Supervisor of Environmental Receiving to ensure information is documented. After review, each log sheet is stamped as reviewed and initialed and dated.

- The labelled samples are transferred to the secured, locked walk-in cooler facility.

- The CompuChem number is listed on the original Chain-of-Custody Form next to the associated client ID when possible.

- The Quiz portion of the CLMS is accessed to produce the worksheets for EPA sample analyses. For EPA samples, the system will generate volatile, semivolatile, and pesticide worksheets. For commercial and inorganic samples, the appropriate worksheets are pulled from the worksheet files; the analysis codes for these samples should have been included with the packing information and confirmed with Customer Service. All laboratory worksheets are distributed to the appropriate Production Planning and Control Planner.

- To produce EPA quality control worksheets for the QC samples associated with a batch of samples, EPA Water or EPA Solid programs of Quiz in the CLMS is accessed and the samples' CompuChem numbers are entered; these worksheets are copied on green paper. To assemble commercial QC worksheets, the appropriate green fraction worksheets are pulled from the trays in the Shipping and Receiving area. Separate QC Sample Records are used to document the analysis of the QC samples associated with a particular system and are put into green QC folders for Report Integration. Included in the commercial folder are the Sample Record (generated by the CLMS), a copy of the order form, and, if necessary, a copy of the Chain-of-Custody Record.

- Commercial file folders are assembled for Report Integration; included in the production sample's folder are the Sample Record, Customer Sample Information Sheet and Chain-of-Custody Record; the QC Sample Record is included in the green Quality Control folder that also goes to Report Integration.

- EPA file folders are assembled for Report Integration; EPA only has the Sample Record in the file folder. A gray envelope contains all materials for the case including: yellow copy of the OTR (Organic Traffic Report), Chain-of-Custody, original air-bill, a copy of the Log-In Sheet, a copy of the EPA Scheduling Log, Custody Tags sealed in plastic bags (if received), and a gray envelope contents sheet (Attachment 9). The white copy of the OTR is returned with a cover sheet to the EPA/SMO (Sample Management Office) (Attachment 10).

- If problems arise concerning received samples, Customer Service is contacted (for commercial samples) or the Technical Management Staff is contacted (for EPA samples).

- Samples are sometimes received from the EPA that should not have been sent. Therefore, they require a transfer. The following steps should be taken:

    1. Fill out a new Chain-of-Custody Form using the information on the sample tags.

    2. Sign the Chain-of-Custody in the first section labeled "Relinquished By."

    3. A Copy of Chain-of-Custody is kept for our records; the original is sent with samples. A copy of the paperwork received with the samples should also be sent.

    4. Notations are made on the Traffic Reports stating samples are being sent to another laboratory.

Samples hand-delivered after business hours should be recorded as follows:

1. The actual date of sample receipt shall be recorded on the Chain-of-Custody (see Note).

2. The date and time of sample receipt are recorded on the TRs as follows:

    a. Organic Traffic Report

    Date and time of receipt in Column F, sample condition upon receipt

    b. Inorganic Traffic Report

    Same as above

The notation "H.D." (Hand-Delivered) or "Received by Common Carrier" should be recorded next to the actual sample receipt time and date. The following calendar day shall be recorded in the block designated for Receipt Date on the Organic/Inorganic Traffic Report.

Samples received after business hours are logged in the same way as samples received during regular business hours. (Page 1 of this SOP details the procedure for logging in samples.)

Note: For New Jersey DEP Chain-of-Custody procedure, see Production Planning and Control SOP 3.2 and the note on page 2 of this SOP.

COMPUCHEM LABORATORIES

CHAIN-OF-CUSTODY RECORD

№ 000933

PROJECT NAME:

PROJECT NUMBER:

SAMPLERS (SIGNATURE)

PRINTED NAME

CLIENT ID (9 CHARACTERS)

1	2	3	4	5	6	7	8	9

#	GC/MS					GC							INORGANICS				OTHER											SAMPLING INFO		REMARKS
No. of Bottles/Vials	624-8240	625-8270	TCL-VOA	TCL-SVOA	Other:	601-8010	602-8020	608-8080	8140	TCL PEST/PCB's	Herbicides	Other:	Metals	Cyanide	TAL Metals	Other:	TOC	TOX	Oil & Grease	Pet Hydro.	Phenols						MATRIX: Water/Soil	DATE	TIME	

Section 1.1
Revision No. 11
Date: June 17, 1991
Page 7 of 20

ATTACHMENT 1

RELINQUISHED BY	Date/Time	RELINQUISHED BY:	Date/Time	RELINQUISHED BY:	Date/Time
COMPANY NAME		COMPANY NAME:		COMPANY NAME:	
RECEIVED BY	Date/Time	RECEIVED BY:	Date/Time	RECEIVED BY:	Date/Time
COMPANY NAME		COMPANY NAME:		COMPANY NAME:	
RELINQUISHED BY	Date/Time	RELINQUISHED BY:	Date/Time	RELINQUISHED BY:	Date/Time
COMPANY NAME		COMPANY NAME		COMPANY NAME:	

SHIPPING INFO

Number of Shipping Containers-

Method of Shipment

Special Handling Requirements

ATTACHMENT 2

Sample Record

Requisition Number: CompuChem Number:
Case: Account Number:
Turnaround: Priority: Due:
Analysis Codes:

Lab Instructions

Receiving Data

Sample Identification: SS Code:
SS Number: Receiving Codes:
Date Received: Time:
Receiving Information:
Containers Received:
Matrix: Temp: Sampling Date(s):

Deliverables

Deliverables Code:

Lab Requirements

Laboratory Completion Date	Repeat Date	
_____	_____	( )
_____	_____	( )
_____	_____	( )
_____	_____	( )

Company Name:

Sample Record

Requisition Number: CompuChem Number:
Case: Account Number:

FOOTNOTES:

Applicable QA Notices:

Company Name:

QUALITY ASSURANCE NOTICE

CompuChem#
Sample ID
Case#
Type of Analysis
Receipt Date

The pH reading for the sample above was ____, the required pH level is ____.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

Preserve In-House
Analyze - Qualify with Notice
Dispose - Client will Resample
Subcontract Lab to Preserve

Supervisor Signature
Date

QANR2
900130

QUALITY ASSURANCE NOTICE

CompuChem#
Sample ID
Case#
Type of Analysis
Receipt Date

The required temperature for Environmental samples requiring Organic/Inorganic Analysis is 4C (+/-2C). The temperature on the sample above was ____.

The Client was contacted by a member of CompuChem's Environmental Marketing Department. The Environmental Receiving Department was instructed to:

Analyze - Qualify with Notice
Dispose - Client will Resample

Supervisor Signature
Date

QANR3
900130

COMPUCHEM LABORATORIES
ENVIRONMENTAL RECEIVING LOG

RECEIVED ON: _____ # OF PAGES _____ OF _____

CLIENT: _____ ORDER# _____ CASE I'D _____

DELIVERED BY: _____ FREIGHT #'s _____ TAT _____ C-OF-C Y or N

SUBCONTRACT# _____ SS# _____

#	REMARKS	GEO	SAMPLE ID	CCC	SOIL	TEMP	SU	CN	OH	PARAL CODES	MX	WATER VOLUME	BLANKS
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													

LOGGED-IN BY: _____ RECEIVED BY: _____ REVIEWED: _____

ATTACHMENT 6

COMPUCHEM LABORATORIES
ENVIRONMENTAL RECEIVING LOG

RECEIVED ON: _____ # OF PAGES _____ OF _____

CASE # _____ ORG/INORG: _____ ORDER# _____ CASE I'D _____

DELIVERED BY: _____ FREIGHT #'s _____ TAT _____ C-OF-C Y or N

ACCOUNT #: _____ CONTRACT # _____ REGION: _____ $ AMT: _____ TAGS Y or N

#	REMARKS	REC	SAMPLE ID	VOL	SOIL	TEMP	COD	CN	PH	ANAL CODES	MX	VOLUME
1												
2												
3												
4												
5												
6												
7												
8												
9												
10												
11												
12												
13												
14												
15												
16												
17												
18												
19												
20												

LOGGED-IN BY: _____ RECEIVED BY: _____ REVIEWED: _____

COMPUCHEM LABORATORIES

date shipped to consignee: _____ date received from consignee: _____

number of sample: _____

consignee name: _____

address: _____

_____

_____

DO NOT REMOVE: FOR COMPUCHEM USE ONLY

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION

At the end of the sampling period, it is vital to ship the sample via express transportation. To insure proper follow-up and prompt analysis, please call 1/800-334-8525 and provide us with the following information:

1. Date Shipped
2. Time Shipped
3. Freight Carrier
4. Freight Bill of Lading Number

Sampling Period

From:

Date _____

Time _____

To:

Date _____

Time _____

Company _____

Address _____

City & State _____ Zip Code _____

Sample Name/Number _____

Return this form in the envelope provided and return with the SAMPLESAVER.

Thank you.

PLEASE NOTE THAT ANY AND ALL PRESERVATIVE(S) ARE TO
BE ADDED BY THE CUSTOMER AT THE TIME OF SAMPLE COLLECTION.

COMPUCHEM

CONTENTS OF FOLDER

DATE: _____

EPA: ( ) ORGANIC ( ) INORG COMMERCIAL: ( )

CASE #: _____ CLIENT: _____

NO. OF TRAFFIC RPTS: _____ ORDER#: _____

#	ITEM	Y	N	NA	COMMENTS
1	CHAIN-OF-CUSTODY	( )	( )	( )	
2	AIRBILL	( )	( )	( )	
3	DAILY LOG SHEET	( )	( )	( )	
4	TRAFFIC REPORT	( )	( )	( )	
5	TAGS	( )	( )	( )	
6	SMO COVER LETTER	( )	( )	( )	
7	SCHEDULING LOG	( )	( )	( )	

PROBLEMS/COMMENTS:

SAMPLE RECEIVING:

DOCUMENTATION CONTROL:

COMPUCHEM LABORATORIES

SAMPLE DELIVERY GROUP (SD)
TRAFFIC REPORT (TR) COVER S

Lab Name: _____ **Contract No.: 68-01-**_____

Lab Code: _____ **Case No.:** _____ **SAS No.:** _____-___

Full Sample Analysis Price in Contract: $_____

SDG No./First Sample in SDG: _____ **Sample Receipt Date:** _____
(Lowest EPA Sample Number in first shipment of samples received under SDG) (MM/DD/YY)

Last Sample in SDG: _____ **Sample Receipt Date:** _____
(Highest EPA Sample Number in last shipment of samples received under SDG) (MM/DD/YY)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1	_____	11	_____
2	_____	12	_____
3	_____	13	_____
4	_____	14	_____
5	_____	15	_____
6	_____	16	_____
7	_____	17	_____
8	_____	18	_____
9	_____	19	_____
10	_____	20	_____

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

_____ _____ _____

Sample Custodian Date

ATTACHMENT 11

SAMPLE LOG-IN SHEET

Lab Name: ______________________ Page ____ of ____

Received By (Print Name): ______________________ Log-in Date: ____________

Received By (Signature): ______________________

Case Number: ____________

Sample Delivery Group No.: ____________

SAS Number: ____________

	Corresponding			
EPA Sample #	Sample Tag #	Assigned Lab #	Remarks: Condition of Sample Shipment, etc.	

REMARKS:

1. Custody Seal(s) — Present/Absent* Intact/Broken
2. Custody Seal Nos.: ________ ________
3. Chain-of-Custody Records — Present/Absent*
4. Traffic Reports or Packing List — Present/Absent*
5. Airbill — Airbill/Sticker Present/Absent*
6. Airbill No.: ________ ________
7. Sample Tags — Present/Absent*
   Sample Tag Numbers — Listed/Not Listed on Chain-of-Custody
8. Sample Condition: Intact/Broken*/Leaking
9. Does information on custody records, traffic reports, and sample tags agree? Yes/No*
10. Date Received at Lab: ________
11. Time Received: ________

Sample Transfer

Fraction: ________ ________

Area #: ________ ________

By: ________ ________

________ ________

* Contact SMO and attach record of resolution

Reviewed By: ______________________

Date: ______________________

Logbook No.: ______________________

Logbook Page No.: ______________________

FORM DC-1

3/90

Form DEP-088
10/86

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTE

SAMPLE ANALYSIS REQUEST FORM

SAMPLE LOCATION

NAME OF FACILITY/ COMPANY/PERSON	CODE	MAILING ADDRESS
SAMPLE LOCATION		

SAMPLE COLLECTOR

SAMPLER(S) Name	DIVISION		
Name	CONTRACT #	Task #	Report Format

SAMPLE IDENTIFICATION

NJDEP SAMPLE NO.	SAMPLING DATE	ABSCISSA (Longitude)
SAMPLE POINT ID	TIME (MIL) SAMPLING BEGAN	ORDINATE (Latitude)
SAMPLE SOURCE CODE	TIME (MIL) SAMPLING FINISHED	ELEVATION
REFERENCE POINT		

SAMPLING PROCESS

COLLECTION METHOD	Code	Other	SAMPLE TYPE	Code	Other
TYPE OF SAMPLER	Code	Other	SAMPLE MATRIX	Code	Other
DESCRIPTION					

FIELD MEASUREMENTS

SAMPLE TEMP. °C	PARAMETER	PARAMETER	PARAMETER
AIR TEMP. °C	VALUE	VALUE	VALUE
WEATHER			

CHAIN-OF-CUSTODY IMPLEMENTED ☐ YES ☐ NO | PRESERVATION CODE(S) ___ ___ ___ ___ ___

REQUESTED ANALYSIS(ES)

PARAMETERS (by Compound or Group of Compounds)	CONTAINER NUMBER	DETECTION LIMIT (digits/units)	EMERG. (Check One)	NON-EMERG.

ANALYSIS(ES) AUTHORIZED BY | TITLE

ANALYZING LABORATORY

CONTRACT LAB NAME	PERSON ACCEPTING SAMPLE	TITLE
LAB ID #	LAB CONTROL #	DATE	TIME (MIL)

COMPUCHEM LABORATORIES

EPA SCHEDULING LOG

WEEK ENDING SATURDAY:

CASE#: ( ) NEW CASE ( ) OLD CASE ( ) COMPLETED CASE

REGION: TURNAROUND TIME:

ACCOUNT #: CONTRACT #: CONTRACT $

****

QUANITY RECEIVED # OF SAMPLES

WATERS	
SEDIMENTS/SOILS	
OTHER	

CONCENTRATION: ( ) LOW ( ) MEDIUM ( ) HIGH

ORDER#:	
AIRBILL#:	

****

PROBLEMS/COMMENTS:

SMO CONTACTED AT (TIME) SPOKE TO:

RESOLUTION FROM SMO:

Documentation Form For:

Revising or Creating Standard Operating Procedures (SOPs): Including Designated Personnel Responsibilities

✓ Revised Procedure _____ New Procedure ✓ Procedure Attached

PPC SOP 1.1: Logging In Samples — 6-17-91
Procedure Area, Title, and SOP Number — Effective Date

Michael Grayson — 6-17-91
Procedure Prepared By — Date

Michael Grayson — 6-17-91
Procedure Read, Understood, and Approved By
Appropriate Laboratory Station Manager — Date

[signature] — 6-18-91
Procedure Read, Understood, and Approved By
Quality Assurance Representative — Date

This procedure(s) meets the requirements as set forth in the following References for Approved Methods:

_____

_____

_____

_____

These procedures describe how tasks are performed in this specific area. If a question arises concerning the proper procedure to follow for an activity in this area, these SOPs should be consulted to resolve the question. Also, these SOPs are a valuable source of material for training purposes.

After the manager of this area believes the person responsible for these tasks has mastered these SOPs, both the manager and the employee should sign and date this form, assuring that these SOPs are understood and will be followed in the daily operations of CompuChem Laboratories. Please forward a copy of this revised or created SOP and a completed form to Quality Assurance.

Employee's name: _____ Date: _____

Employee's title: _____

Employee's name: _____ Date: _____

Employee's title: _____

Manager's name: _____ Date: _____

Manager's title: _____

WARRZYN'S TAL INORGANIC LOW LEVEL SOP's.

APPENDIX A

Effective Date: 7-2-91

ATOMIC ABSORPTION SPECTROMETRY
Furnace - Direct Injection

Scope and Application: Metals in solution can be readily analyzed by Atomic Absorption Spectrometry using either flame, furnace or hydride techniques. The furnace - direct injection technique allows for lower detection limits. The use of the graphite platform in furnace analyzed can improve sensitivity and reduce some matrix interferences.

Method: Furnace; direct injection

Reference: EPA 1984, Section 200
Analytical Methods for Zeeman Graphite Tube Atomizers - Varian 1986
Spectra AA - 300/400 Zeeman Operation Manual - Varian March 1988

Sample Handling: Acidify with concentrated nitric acid to ph < 2. Drinking waters and filtered groundwater samples free of particulate matters and organics may be analyzed directly, while wastewaters, leachates, solids, etc., must be digested prior to analysis (refer to appropriate digestion procedures). Samples must be analyzed within 6 months.

Reagents and Apparatus:

1. Zeeman Automatic Absorption Spectrometer - 400
2. Zeeman Graphite tube Atomizer
3. IBM Personal System/2 Model 30 Computer
4. EPSON EX-800 Printer, Citizen HSP-500 printer, or similar adaptable printer.
5. Required metal lamp and power source
6. Stock and standard solutions for required metal
7. Class A volumetric glassware
8. Instr-analyzed nitric acid
9. Deionized (D.I.) water
10. Argon gas - prepurified grade
11. Graphite partition tubes
12. Graphite plateau tubes and platforms
13. Disposable 2 mL sample cups
14. Eppendorf 100-1000 microliter pipetor
15. Disposable 10 ml beakers

Procedure:

Power Up Procedure

1. Turn on argon gas and cooling water.

2. **Always turn the system on in the following order:** spectrometer, furnace, printer, and computer. This initializes the communication relays correctly so that all components of the system can "talk" to each other.

3. After the DOS prompt has been displayed, type "Zeeman" and press Enter. After a brief pause, an introductory message will then be displayed followed by the PROGRAM MODES page. Follow the on-screen instructions to select the appropriate mode.

Automatic Run Using the Sampler:

Notes:

a. Only programs which have been stored can be used for an automatic run.

b. For all programs, the method of sample introduction (**instrument parameters** page) must be specified as **sampler automixing** (for automatic mixing of calibration standards from a single, high concentration standard) or **sampler premixed** (for a full set of calibration standards that are prepared by the operator prior to analysis).

c. Options on the **report format** page allow raw data to be printed either as it is collected during an analytical run (used for most analyses), or after the analysis is completed (used for sequential runs of multiple elements).

d. If an automatic run is stopped and then restarted, the sampler will automatically perform a tube clean and analyze a blank. It will then continue on according to the instructions set in the **sequence control** page.

e. F9 through F12 are hard keys with their function on the supplied overlay. F1 through F6 are soft keys; their functions will change from one page to the next. The function for each soft key is displayed at the bottom of the screen and only those displayed are active for that page.

f. Any page described below can be recalled by returning to the **index** and entering the appropriate page number.

1. Perform daily maintenance. Check the condition of the graphite tube and replace as necessary.

2. From the **program modes** page, press **automatic run.** The system will automatically display the **sequence selection** page.

3. On the **sequence selection** page, press F1 to clear the sequence of previous element(s) and enter the code of the program to be run. If more than one program is to be run, press **enter** after each element program number. Press F6 to recall program. The **sequence control** page will automatically be displayed.

4. Follow on-screen instructions to enter the number of initial tube burns for cleaning (1 or 2 for previously used furnace tubes, 3 or 4 burns for new tubes), the starting position for the run (usually position #1), and the last position for the run. Note that when the analysis at the last position is completed, the automatic run is terminated and the element lamp is shut off automatically. Setting the final position to leave several empty cups at the end of the analytical run allows necessary repeats or

dilutions to be added to the end of the current run, saving lamp warm-up and calibration time.

5. Return to the **index** and select page 6 **(optimization)**

   a. Open the lamp turret cover and ensure that the required lamp is in the operating position.

   b. Observe the signal bar labelled **align hc lamp** displayed on the video screen. Turn the horizontal lamp base adjusting screw (the top one of the two) fully clockwise. Now turn this screw slowly counter-clockwise until the first peak is detected (the length of the signal bar will increase). Continue adjusting this screw until the length of the signal bar is the maximum obtainable (if the signal bar is fully extended, press the rescale soft key, F1, to bring the signal bar back on scale and again adjust the screw to obtain maximum signal. Note particularly that turning the horizontal adjusting screw further counter-clockwise may produce a second peak. **Do not** align the lamp on this second peak - **always** align the lamp on the first peak. Carefully adjust the vertical adjusting screw (the bottom one of the two) so that the length of the signal bar is the maximum obtainable (if necessary, press F1 to rescale the signal bar).

   c. Record the photomultiplier voltage in the instrument log book. A constantly increasing voltage over time is evidence of decreasing efficiency of the element lamp. Monitor this voltage to determine when element lamps should be replaced.

   d. When switching from partition to platform tubes (or vice-versa), check the position of the graphite tube automizer:

      Hold a piece of white card between the righthand end of the graphite tube automizer and the sample compartment window. Use the furnace vertical adjust and position the automizer until light from the hollow cathode lamp is obviously passing through the graphite tube on to the card.

      Remove the card. Observe the signal bar labelled **align hc lamp** displayed on the video screen. Use the furnace vertical adjust and carefully adjust the position of the graphite tube automizer until the length of the signal bar is the maximum obtainable.

6. Use the soft key indicated, or return to **index** to select **standards page.** This page tells which standards are to be used for calibration.

7. Use the soft key indicated, or return to **index** to select **sampler page.** This page lists the volume of standards, blanks, samples and modifier that are to be used for analysis.

8. At the **sampler** page, press F2 to align the sampler arm. Place a finger on the arm as it starts to descend into the furnace and gently lower the arm by hand. Carefully adjust the sampler position using the two adjustment knobs on the base of the autosampler so that the capillary is exactly in the center of the sample injection hole. With the capillary down in the furnace, and using the mirror, turn the height adjusting screw so the capillary is about 1 mm above the bottom of the tube or platform.

9. Return to **index** and select **report format** page. Enter operator initials, analysis date, and batch number. Review the defaults set for the remaining parameters. Follow the on-screen instructions for using the **home** key to make any needed changes.

10. If sample labels are to be printed with the raw data, press F6 and enter appropriate labels. Note that the **Tab** key will jump the cursor to the next field; the ↑ and ↓ keys to move the cursor up and down the columns.

11. Press F10 to zero the instrument before beginning analysis. Press F11 to start the automatic run.

12. To change basic operating conditions (these are default conditions recalled automatically with the analytical program), press F12 to pause run, return to the **index** and select page 4, **instrument parameters.** Parameters may be changed using the **home** key or soft keys as indicated. Press F11 to resume the analytical run.

Furnace Maintenance:

The following maintenance is to be done each day the furnace is operated:

1. Clean the furnace windows.

    a. Twist out furnace windows from furnace unit.
    b. Wipe windows with a Q-tip moistened with alcohol.
    c. Rinse windows with D.I. water and dry with a Kim-Wipe
    d. Re-insert windows in furnace.

2. Check machine windows and clean if needed.

3. Wipe inside of furnace with a Q-tip moistened with alcohol.

4. Fill the autosampler rinse bottle with D.I. water.

5. Open the syringe compartment door on the autosampler and pull the syringe assembly carefully out of its mounting. Remove the plunger from the syringe, and on the **sampler** page, press F3 to rinse the syringe and bleed any air bubbles from the syringe. Press F3 and rinse again, while water is dripping from syringe insert the plunger into the syringe. Wipe the syringe dry and carefully re-insert in its mounting.

6. **Inserting graphite tube**

    a. Swing toggle level on top of furnace fully clockwise to open furnace.

    b. Place graphite tube in the graphite shroud in the center block. Align sample introduction part of the graphite tube with the opening in the furnace block.

    c. Swing the toggle lever fully counter-clockwise and the righthand electrode assembly will automatically close on the center block.

d. Before using a new graphite tube for analyses, use the tube clean utility (**signal graphics** page) 3-4 times to remove any contamination. This can be done automatically by entering 3 or 4 tube cleans in the appropriate field on the **sequence control** (page 11) before starting an analytical run.

Effective 7-2-91

ATOMIC ABSORPTION SPECTROMETRY FLAME-DIRECT ASPIRATION

Scope and Application: Metals in solution can be readily analyzed by Atomic Absorption Spectrometry using either flame or furnace techniques. The flame-direct aspiration can be used for most metals but is generally not as sensitive as the furnace method. Both the air-acetylene and nitrous oxide-acetylene flame techniques are described in this operating procedure as well as the use of emission spectroscopy.

Method: Flame; direct aspiration

Reference: EPA 1984, Section 200

"Analytical Methods for Flame Spectrophotometry", Varian, 1979

Spectr AA - 10/20 Operation Manual, Varian

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Sample Handling: Acidify with concentrated nitric acid to pH<2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Samples must be analyzed within 6 months from sampling date.

Reagents and Apparatus:

1. Varian Spectr AA-20
2. Stock and standard metal solutions
3. class A volumetric glassware
4. Instra-analyzed nitric acid
5. Deionized (D.I.) water
6. Hollow cathode element lamps
7. Disposable 10 mL beakers
8. Eppendorf 100-1000 uL pipetter
9. Oxford 5 or 10 ml pipetter
10. Acetylene gas
11. Air supply
12. Nitrous oxide gas
13. Air-acetylene burner head or nitrous oxide-acetylene burner head

Setup:

1. Power on instrument. The computer will automatically start with a memory check. When the first screen appears, it is ready to operate.

Note: Allow instrument a 1/2 hour warm up period for electronic and optical components to achieve thermal equilibrium before beginning analysis.

2. Power on printer. Check the paper supply.

3. Install the desired element lamp in the lamp turret by depressing the middle white button behind the socket, inserting the lamp, and releasing the button. Ensure that the lamp is secure and that the connections are fitting properly.

   Note: Allow lamp a 10-15 minute warm up period before beginning analysis.

Procedure:

This procedure will outline an analysis as it would be run following the instructions given on sequential computer screens. **Note:** Any time during setup the "Index" key can be used to go to any screen in the software.

1. Soft key selections allow the operator to develop program, modify program, or automatic run. The typical analysis will be run by selecting "Automatic Run."

   Note: After completing required information on the present screen use the soft keys to call up the next screen.

2. **"Sequence Selection".** This screen lists the programs on file. Use the "Clear Sequence" soft key to erase the last sequence used, type in the number corresponding to the program desired, and press "Sequence Selection" soft key. This will automatically recall the program.

3. **"Sequence Control".** The screen is used for autosampler control only. Go to next screen by pressing "Report Format" soft key.

4. Use cursor arrows and numeric keys to enter operator and date. The "Home" key is used to change entries of other parameters.

5. **"Sample Labels".** Use the cursor arrows and numeric keys to enter labels.

   Note: Sample labels will only be printed if the automatic run is used.

6. **"Optimization".** This screen is used to optimize wavelength and lamp position.

   a. Ensure lamp is located correctly and is on (lamp is automatically turned on when program is called up).

   b. Select proper slit width.

c. Release brake ("off") and set approximate wavelength. Set brake ("on") and fine-tune the wavelength to achieve maximum intensity on HCl bar graph. "Rescale" (soft key) as often as necessary to keep graph on scale.

d. Optimize lamp position by adjusting the adjusting screws on back of the lamp socket. Adjust for maximum intensity on the bar graph. "Rescale" as often as necessary.

e. If background is used, adjust maximum intensity on background bar graph by 2 set screws on the background corrector housing. Set attenuator ("In" or "Out") if necessary. "Rescale" if necessary.

f. Record the photomultiplier voltage in the instrument log book. A constantly increasing voltage over time is evidence of decreasing efficiency of the element lamp. Monitor this voltage to determine when element lamps should be replaced.

Note: HCl and background lamp intensities should match as closely as possible. The attenuator will cut down background intensity. A lower lamp current will lower its intensity.

7. **Flame Ignition**

a. Turn on compressed air to 50 psi (35-65 psi)

b. Turn on acetylene tank, pressure should be 7-15 psi.

c. Turn on nitrous oxide tank (if necessary - the proper burner head must be in place for ignition to occur). Tank pressure should be 50 psi (35-65 psi).

d. Press "Ignite" key and hold down until flame ignites.

Note: Let burner head warm to equilibrium before analysis; 5 to 10 minutes for an air-acetylene flame, 10 to 15 minutes for a nitrous oxide-acetylene flame.

8. **Signal Optimization**

a. Press "Signal Optimization" soft key on optimization screen.

b. Adjust burner head using 2 adjusting screws and rotation lever for maximum intensity while aspirating a high standard.

c. Adjust the nebulizer/glass bead by slowly turning the screw directly below the nebulizer.

9. **Flame Emission Procedures**

   a. In this method, no element lamp or background correction is used. Burner head position and wavelength are optimized while aspirating the highest working standard.

   b. Turn the burner head full right or left (approximately 30° angle).

   c. Select optimization screen.

   d. Adjust wavelength for maximum intensity.

   e. Press "Emission Setup" soft key.

   f. Continue with automatic/non-auto run.

10. **Automatic Run (no autosampler)**

   Note: Only pre-existing programs can be used.

   a. Press "Start" key to initialize run. Once a run is started, it can be paused by pressing the "Stop" key, but none of the program parameters can be changed.

   b. Press "Instrument Zero" key after program has been recalled to establish a zero instrument baseline.

   c. Aspirate standards or sample and press "Read". The instrument will display the std #/sample # on the top of the screen, along with the absorbance.

   d. The "Previous Sample"/"Next Sample" soft keys can be used to repeat a specific analysis or move ahead in the sample order, "solution type" can be used to restandardize by starting at "blank".

   e. If more than 66 samples and standards are to be run, add them at the end of the run and depress "Previous Sample" key for each sample. Since the sample labels cannot be changed, leave the last few labels blank on the sample labels page, and write them in when the run is completed.

   f. Press "Stop" key to pause or end the analysis.

11. **Non-automatic Run**

   Note: A modified or newly developed program can be run in this mode, a well as a pre-existing program.

   a. Set up instrument according to previous instruction. Note that the sample labels and report format cannot be printed in this mode.

b. Advance to "Standards" screen by use of soft key on optimization screen or through the "index".

c. Aspirate standards/samples and press "Read" key as in the automatic run.

d. This mode is not limited to the samples. As no labels are printed, these must be written onto the printout by hand.

12. **Instrument Shut Down**

a. Turn off flame ("Flame Off" key).

b. turn off all gases.

c. Recall program #10 or # (Emission programs), so that no lamp is turned on unnecessarily when the instrument is not turned on.

d. Turn off printer.

e. Turn off instrument.

[rff-metcont-200]

Effective: 6-2-91

INDUCTIVELY COUPLED PLASMA - ATOMIC EMISSION SPECTROMETRIC METHOD

Scope and Application: Metals in solution can be readily analyzed by atomic emission using an inductively coupled plasma. Dissolved metals are determined in filtered and acidified samples. Total metals are determined in acidified, but unfiltered samples. Appropriate steps must be taken in all analyses to ensure that potential spectral interferences are taken into account.

Method: Inductively coupled plasma - atomic emission.

Reference: "Methods for Chemical Analysis of Water and Wastes", Method 200.7 EPA 1984.

"Inductively Coupled Plasma - Atomic Emission Spectroscopy", Method 6010, SW-846, November 1986.

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

"Instructions: Plasma 40 Emission Spectrometer", Perkin-Elmer, 1987.

Sample Handling: Acidify aqueous samples with concentrated nitric acid to pH <2. All samples must be digested prior to analysis (refer to appropriate digestion procedure). All samples must be analyzed within 6 months of sampling date.

Reagents and Appartus:

1. Plasma 40 Perkin-Elmer ICP Spectrometer
2. Argon (liquid: "high purity" or gaseous: "prepurified" grade)
3. Stock and intermediate metal standard solutions
4. EPA, ERA, or other reference standard solutions
5. Nitric acid, conc. (instra-analyzed or equivalent grade)
6. Class A volumetric glassware
7. Deionized water
8. Disposable 15 mL centrifuge tubes
9. 100 uL Eppendorf pipetter
10. 5 or 10 mL Oxford pipetter
11. Yttrium or Scandium stock solution
12. IBM AT Computer or equivalent
13. Epson 800 printer

Procedure:

Instrument Set-Up Procedure for Plasma 40:

1. Turn ON power switch if necessary (routinely left ON throughout week). Allow 1 hour for RF generator to warm up and electronic and optical components to achieve thermal equilibrium.

2. Perform daily maintenance as specified in Maintenance Procedures: check pump, pump tubing, and nebulizer tips for wear, cleanliness, etc.

3. Turn on argon at tank. The first three indicator lights on the ICP (Power, RF ready, Interlock) should light.

4. Lock pump tubing in place, raise torch to the "ignite" position, and press "RF on".

5. When plasma ignites, lower torch to the run position (the injector tip should be even with or just below the bottom of the lowest RF coil).

6. Turn on pump and aspirate rinse water*. Allow plasma to stabilize 30 to 40 minutes before starting analysis.

Computer Start-Up Procedure:

1. Turn computer and printer power on (the computer will automatically start with a memory check).

2. Type CD ICP and press Return to enter the ICP directory. Then type ICP and press Return again to load software (approximately 10-15 seconds).

3. Perform a BEC check as specified in Maintenance Procedures. The BEC and CV values must be within the specified range before any analysis is done.

Sample Analysis:

1. Before starting analysis, for each element to be analyzed:

   a. Press F1 to select the Element mode, type the appropriate element file name and press Alt F9 to retrieve it from Library.

   b. Press F8 to select Spectrum mode.

   c. Analyze a single element standard at approximately 2-10X the IDL.

   d. Analyze the ICS AB solution.

   e. Analyze 1-3 samples representative of the digestion set.

   f. Compare the displayed spectra to check for spectral interferences. Reset background correction points as needed. If there are overlapping peaks or other spectral interferences present, an alternate wavelength or interelement correction must be used.

   g. Press F8 to leave the Spectrum mode. If wavelength calibration or background correction points were changed, press F9 to save the changes.

* Rinse water should be D.I. water with a small amount of liquid detergent (such as Liquinox or Whisk) added to improve wetting of tubing and spray chamber. Approximately 1-2 mL of soap per 500 mL water should be sufficient.

2. To store a list of sample labels to be used for the analytical run, select Report mode (F3), then ID/Wt mode (F8). Enter a file name, type in N (no) in the field for raw emission counts, mg/L for uncorrected units, and leave the corrected units field blank. Enter sample labels in the sample ID field in the exact order of analysis; include all check standards, QC samples, etc. If it is possible that additional samples may be added to the end of an analytical run (dilutions, post-digestion spikes, linear range standards, etc.) add additional sample labels to the ID/Wt file in the form of single letters (A, B, C, etc.) and manually write in the correct sample labels after the analytical run is completed. Alternatively, a new ID/Wt file may be created **after** the analytical run is completed (in this case the raw data must be reprinted with the new file by selecting Report Format 1 (F5) in the report mode and responding to the prompts). Save ID/Wt files by pressing F9 (to library).

3. Press F2 to select the Method mode.

4. Type the method file name and press Alt F9 to retrieve the desired method panel from Library, or create a new panel using existing element files. Standard conditions are 35 second read delay, 2 replicates per sample, report format #2 and a data file name composed of the date (mmdd) and a sequential letter identifier (e.g. 0123B for the second analytical run on Jan 23). An internal standard (usually yttrium) must be included in any method. Background correction points are already included in each element file.

5. Add yttrium (or scandium) stock solution (1000 mg/L) as an internal standard to all standards, blanks, and samples in a ratio of 0.1 mL yttrium stock to 10 mL sample. This allows automatic correction for matrix differences in viscosity, surface tension, etc.. If the autosampler is to be used, samples can be pipetted directly into 15 mL centrifuge tubes. Otherwise mix sample and yttrium in small disposable beakers.

If autosampler is used:

1. If the autosampler is to be used, load sampler starting with the calibration standards in order of decreasing concentration (highest concentration first, calibration blank last).

2. Start automatic run (F5). Respond to the prompts that appear at the bottom of the screen:

   a. "Press start function key to begin this analysis": press F5

   b. "Enter ID/Wt file": type ID/Wt file name and press Return.

   c. "Do you wish to rinse between tubes (Y or N)": type Y and press Return. N may be selected only for clean samples where no carry-over problems are anticipated. **Always** rinse between samples when analysis is following CLP protocols, or analyzing for Sb, Cr, or Zn.

d. "Enter position of the last sample in tray": type appropriate number and press Return (you may wish to enter a number several positions past the last sample to allow room for the addition of necessary dilutions, etc. at the end of the run).

e. "Do you wish to re-standardize (Y or N)": type N or Y and press Return. N is usually selected. Y will allow restandardization of the instrument during an automatic run but additional autosampler positions will be unavailable for samples. If Y is selected, additional on-screen instructions will prompt for position of additional calibration standards.

f. "Do you wish to wavelength calibrate during the analysis? (Y or N)": type N or Y and press Return. N is usually selected. Y will allow recalibration of all wavelengths used in the current method before analysis is started but additional autosampler positions will be unavailable for samples. If Y is selected, additional on-screen instructions will prompt for position of additional wavelength calibration standards. The system will then begin the analysis.

3. When the analysis is complete press F2 to select Method mode before exiting software to ensure the data file is stored permanently. Then set up the next panel, return to Report mode to set up a new ID/Wt file or reprint data, or press ESC to exit the ICP software.

If samples are to be run manually:

1. Press F2 to start a manual run and respond to prompts to calibrate instrument: Press F6 (Standard), aspirate the first calibration standard and press Return. At the prompt, aspirate the next standard(s) and press Return. When all calibration standards have been analyzed press F5 (Blank) aspirate the calibration blank and press Return. This completes the instrument calibration.

2. To analyze samples, type in sample label if needed, press F7 (Sample), aspirate sample and press Return. Repeat with all samples in the run.

Computer Shut-Down Procedure:

1. When analysis is complete press F2 (Method mode). At the message "Do you wish to quit method"? Type Y.

2. Press "ESC". At the message "Do you wish to quit method?" Type Y.

3. Turn off computer power switch.

4. Turn off printer.

Caution: Never turn off computer power while still using ICP software. This can cause partial loss of files and other errors.

Instrument Shut-Down Procedure:

1. Aspirate a dilute nitric acid solution (approx. 10%) for 1 to 2 minutes to clean sample introduction system.

2. Aspirate D.I. water for 5 minutes to rinse system thoroughly.

3. Turn off pump and release pump tubing.

4. Press "RF off" to extinguish plasma.

5. Shut off argon flow at tank.

6. If the ICP **will not** be used for **2 days or more**, turn off ICP power switch. Otherwise, leave the ICP power **ON.**

Quality Control:

1. Establish a standard curve with the appropriate calibration standards plus a blank. Record the emission count for the internal standard in the ICP log book. The emission count should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check pump tubing, nebulizer tips, nebulizer flow, wavelength calibration, etc.).

2. **The first analyses for each analytical run are, in order:**

   a. Initial calibration verification standard (ICV)

   b. Initial calibration blank (ICB)

   c. Initial standard at 2X the CRDL (CRI). **Note:** The CRI is not necessary for Ca, Mg, Na or K.

   d. Initial interference check sample, solution A (ICSA).

   e. Initial interference check sample, solution AB (ICSAB)

   f. Laboratory control standard - an ERA, EPA, or other reference standard digested with the sample set (LCS)

To continue with sample analyses, the ICV must be within 90-110% of the true value, the ICB must be less than the CRDL, and the LCS and ICS solutions must be within 80-120% of the true value. If these QC criteria are not met, discontinue the analytical run and perform necessary troubleshooting.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are to be analyzed, a duplicate and spike are still required. Duplicates and spikes are to be within required control limits or the data must be flagged appropriately (N for spikes, * for duplicates). Additionally, if a digested spike is outside required control limits, a post-digestion spike must be analyzed for that sample.

4. For each sample batch (same matrix and project) one sample must be analyzed at an additional 5X dilution for the ICP serial dilution analysis (L). If the original sample concentration is at least 50X above the IDL, the serial dilution must agree within 10% of the original sample concentration or data for all associated samples must be flagged appropriately (E).

5. A continuing calibration verification standard (CCV) and blank (CCB) are to be analyzed, at a minimum, after every 10 analyses. If less than 10 analyses are performed, a CCV and CCB are still required. The last samples analyzed in the run are to be a CCV and CCB. The CCVs must be within 90 - 110% of the true value or the samples run after the last acceptable calibration standard are to be reanalyzed.

6. At the end of each analytical run, but before the final CCV and CCB, the CRI, and ICS solutions A and AB are to be reanalyzed. The ICS must be within 80-120% of the true value or the samples run after the last acceptable calibration verification standard are to be reanalyzed.

7. Refer to the appropriate Quality Assurance Project Plan (QAPP) for project specific QC information (additional QC requirements, matrix spike and duplicate control limits, etc.).

8. Detection limit verifications and linear range analyses must be performed each quarter. Interelement correction factors are to be determined annually at a minimum. Interelement correction factors must be recalculated on an analyte and wavelength specific basis any time background correction points are changed in an element file. Additionally, for greatest accuracy, interelement correction factors should be re-determined for any analytical batch that is expected to have high concentrations of common interferents (e.g. any soil, sediment, sludge, or leachate matrix).

Daily Maintenance Procedures - Plasma 40

1. **Pump rollers:** With the pump on, feel along the bottom of the pump to determine that all the rollers are turning smoothly with no resistance or pulling. If a "sticky" roller is found a service call must be placed to Perkin-Elmer to correct the problem. A sticky roller will cause rapid deterioration of pump tubing resulting in erratic results.

2. **Pump tubing:** Check pump tubing for excessive stretching, soft or flattened spots. This can cause irregular or diminished sample flow resulting in reduced sensitivity and lack of precision in sample results. When pump tubing is changed, (usually after 6-8 hours of use) it is necessary to trim ends of the new tubing so the length from the black stops to the end of the tubing is kept constant. Failure to trim tubing ends can cause imprecise results due to a longer sample read delay.

3. **Nebulizer tips:** Remove nebulizer end cap and check nebulizer tips visually and with the cleaning wire for clogs, salt build-up or other deposits. Follow the instructions in the Plasma 40 operating instructions for replacing nebulizer tips if necessary (Part 2, pg 3-8). Used tips may be cleaned by soaking overnight in 10% nitric acid followed by thorough rinsing with D.I. water. Finally, with the argon on, aspirate water and observe the spray pattern. The nebulizer should produce a fine, even mist with no large droplets with the direction of the spray approximately perpendicular to the face of the end cap (should not deviate more than about 20°). If the spray pattern looks uneven, "bent", or is pulsing excessively, recheck pump tubing and review nebulizer maintenance to correct the problem.

4. **BEC check:** This is an indication of how well the ICP system is performing. After the plasma has been ignited and allowed to stabilize for 30-40 minutes perform the following steps:

   a. At the DOS prompt type ICP and press Return to load software.

   b. Type MnBEC and press Alt F9 to retrieve this method from Library. Press F6 to start a manual run.

   c. Press F6 again (standards), aspirate a 1.0 mg/L Mn standard and press Return. The ICP will analyze 10 replicates of this standard. The Coefficient of Variance (CV) for these readings should be <2.0. If a higher values is obtained a sample introduction or instrument calibration problem is indicated. Check pump tubing and wavelength calibration for Mn and repeat the analysis. Record the CV in the maintenance log book.

   d. Press F5 (blank), aspirate a blank, and press Return to complete the calibration.

   f. Turn off the torch (RF off), aspirate D.I. water, and press F7 (sample). The resulting concentration should be $\leq |0.040|$. If a higher value is obtained, a problem with the sample introduction system is indicated. Review maintenance and, if the problem cannot be corrected, place a service call with Perkin-Elmer. Record the BEC in the maintenance log book.

   i. Re-light the torch and press ESC to end the manual run. Allow the plasma to stabilize 10-15 minutes before beginning any analysis.

5. **Final rinse:** When analysis for the day is complete, aspirate dilute (approx. 10%) nitric acid for one or two minutes followed by D.I. water for approximately 5 minutes. This will help prevent deposits from building up in the sample introduction system. Remember to release pump tubing when completed.

Weekly Computer Backup:

1. Once a week data files should be copied to floppy disks and deleted from the hard disk. Data files on floppy disks should be saved for one year.

2. Periodically (every 1-3 months depending on work volume), files should be reviewed, old files deleted and the entire system backed-up.

Other Maintenance:

1. Occasionally, additional maintenance will be necessary to correct problems arising from time and wear on the system. Any additional maintenance performed (including P.E. service calls) should be listed in the maintenance logs. These include periodic cleaning of the torch assembly, inspection of O-rings in torch assembly, and wavelength recalibration. Generally, these procedures will only be performed in response to observed problems. Refer to the Plasma 40 operating manual for specific directions.

ICAP CALIBRATION STANDARDS

Element	Wave-length	Detection Limit(ug/L)	Cal. Std. 1 (ug/L)	Cal. Std. 2 (ug/L)	Cal. Std. 3 (ug/L)	ICV (ug/L)	CCV (ug/L)
Al	237.335	50	20,000	400	---	2500	4000
Al	396.152	50	20,000	400	---	2500	4000
Sb	206.833	50	2000	500	250	1000	1000
Ba	233.527	10	10,000	200	---	500	2000
Be	313.107	5	1000	20	---	250	200
Cd	228.802	5	1000	50	---	500	200
Cd	214.438	5	1000	50	---	500	200
Ca	317.933	1000	200,000	10,000	---	10,000	80,000
Cr	267.716	10	10,000	500	200	1000	2000
Cr	205.552	10	10,000	500	200	1000	2000
Co	238.892	50	10,000	200	---	2500	2000
Co	228.616	10	10,000	200	---	2500	2000
Cu	324.754	10	10,000	100	---	1000	2000
Cu	224.700	20	10,000	100	---	1000	2000
Fe	238.204	20	20,000	200	---	1000	4000
Pb	220.353	100	10,000	500	---	5000	2000
Pb	216.999	100	10,000	500	---	5000	2000
Mg	285.213	1000	100,000	5000	---	10,000	40,000
Mn	257.610	10	10,000	100	---	500	2000
Ni	352.454	20	10,000	100	---	1000	2000
Ni	232.003	20	10,000	100	---	1000	2000
Ag	338.289	10	1000	---	50	500	200
Na	330.237	2000	100,000	5000	---	20,000	40,000
Sn	189.989	200	10,000	1000	---	2500	5000
V	292.402	50	10,000	500	250	2500	2000
Zn	213.856	10	10,000	100	50	500	2000

ICAP CALIBRATION STANDARDS

ICAP calibration standards are prepared from both multi-element stock solutions purchased from SPEX Industries (custom mixed standards) and single element stock solutions from VWR and Baxter (Ricca or Mallinckrodt as available).

XWE-1	XWE-2	XWE-3a	XWE-4a
2000 mg/L Fe	20,000 mg/L Ca	1000 mg/L Cr	2000 mg/L Al
1000 mg/L Cu	10,000 mg/L Mg	1000 mg/L Pb	1000 mg/L Ba
1000 mg/L Mn	10,000 mg/L Na	1000 mg/L V	1000 mg/L Co
1000 mg/L Ni		100 mg/L Cd	100 mg/L Be
1000 mg/L Zn			100 mg/L Ag

XWE-6a
500 mg/L Pb
250 mg/L Co, Al
100 mg/L Cu, Ni, Fe, Cr
50 mg/L Ba, Cd, Ag, Zn, Mn

Single Element Stock Solutions 1000 mg/L

Sb	Ag
Be	Na
Ca	V
Cr	Zn
Mg	Sn

Calibration Standard #1:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, Zn:** Into a 1 L volumetric flask, add 500 mL of de-ionized (D.I.) water and 50 mL of concentrated HCl. Pipet 10 mL each of XWE-1, XWE-2, XWE-3a, and XWE-4a. Dilute to volume with D.I. water.

2. **For Sb:** Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 1.0 mL of 1000 mg/L Sb stock solution. Dilute to volume with D.I. water.

3. **For Sn:** Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 5.0 mL of 1000 mg/L Sn stock solution. Dilute to volume with D.I. water.

Calibration Standard #2:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Na, V, and Zn:** First prepare 10X dilutions each of XWE-1, XWE-2, XWE-3a, and XWE-4a. Then, into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 1.0 mL of XWE-1 (10X dilution), 5.0 mL of XWE-2 (10X dilution), 5.0 mL of XWE-3a (10X dilution), and 2.0 mL of XWE-4a (10X dilution). Dilute to volume with D.I. water.

2. **For Sb:** Into a 200 mL volumetric flask, add 100 mL of D.I. water and 10 mL of concentrated HCl. Pipet 50 mL of Sb Calibration Standard #1 and dilute to volume with D.I. water.

3. **For Sn:** Into a 200 mL volumetric flask, add 100 mL of D.I. water and 10 mL of concentrated HCl. Pipet 20 mL of Sn Calibration Standard #1 and dilute to volume with D.I. water.

Calibration Standard #3:

1. **For Cr, Ag, V and Zn:** First prepare intermediates as follows:

   50 mg/L Ag and Zn: Into a 100 mL volumetric flask, add 10 mL of 1:1 HCl. Pipet 5.0 mL each of single element Ag and Zn stock solutions and dilute to volume with D.I. water.

   100 mg/L Cr: Into a 100 mL volumetric flask pipet 10.0 mL of single element Cr stock solution. Add 5 mL of 1:1 HCl and dilute to volume with D.I. water.

   50.0 mg/L V: Into a 100 mL volumetric flask pipet 5.0 mL of single element V stock solution. Add 5 mL of 1:1 HCl and dilute to volume with D.I. water.

   Then, into a 500 mL volumetric flask, add 250 mL D.I. water and 25 mL of concentrated HCl. Pipet 1.0 mL of 100 mg/L Cr intermediate, 0.5 mL of 10 mg/L Ag-Zn mixed intermediate, and 2.5 of 50 mg/L V intermediate. Dilute to volume with D.I. water.

2. **For Sb:** Into a 100 mL volumetric flask, add 50 mL of D.I. water and 10 mL of 1:1 HCl. Pipet 50 mL of Calibration Standard #2 and dilute to volume with D.I. water.

Initial Calibration Verification:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, and Zn:** Into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 10 mL of XWE-6a, 10 mL of 1000 mg/L Mg stock, 10 mL of 1000 mg/L Ca stock, and 20 mL of 1000 mg/L Na stock solutions. Dilute to volume with D.I. water.

2. **For Sb:** Into a 500 mL volumetric flask, add 250 mL of D.I. water and 25 mL of concentrated HCl. Pipet 0.5 mL of 1000 mg/L Sb stock and dilute to volume with D.I. water.

3. **For Sn:** Into a 1 L volumetric flask, add 500 mL of D.I. water and 50 mL of concentrated HCl. Pipet 2.5 mL of 1000 mg/L Sn stock and dilute to volume with D.I. water.

Continuing Calibration Verification Standard:

1. **For Al, Ba, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, Ag, Na, V, and Zn:** Into a 1 L volumetric flask, add 500 mL D.I. water and 50 mL of concentrated HCl. Pipet 2.0 mL each of XWE-1, XWE-3a, XWE-4a, and 4.0 mL of XWE-2. Dilute to volume with D.I. water.

2. **For Sb:** Use Sb Initial Calibration Verification Standard.

3. **For Sn:** Into a 200 mL volumetric flask, add 100 mL D.I. water and 10 mL of concentrated HCl. Pipet 1.0 mL of 1000 mg/L Sn stock and dilute to volume with D.I. water.

Effective: 7-2-91

ACID DIGESTION FOR AQUEOUS SAMPLES AND EXTRACTS ICP/Flame-AA

Scope and Application: This acid digestion is applicable to all aqueous sample matrices. A nitric/hydrochloric acid digestion is used to prepare all samples which are to be analyzed by flame atomic absorption spectroscopy (flame-AA) or by inductively coupled plasma spectroscopy (ICP). A nitric acid/hydrogen peroxide digestion is used to prepare samples for analysis by graphite furnace atomic absorption spectroscopy (GFAA).

Method: Nitric acid/hydrogen peroxide and nitric/hydrochloric acid digestions

Reference: "Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Sample Handling: Aqueous samples must be acidified with concentrated nitric acid to pH <2. Set up digestion as soon as possible; digested sample must be analyzed within 6 months.

Reagents and Apparatus:

1. Hot plate
2. 250 mL beakers
3. 100 mL graduated cylinders
4. Class A volumetric glassware
5. Deionized (D.I.) water
6. Instra-analyzed nitric acid, or equivalent
7. Distilled nitric acid (GFAA digestion only)
8. Instra-analyzed HCl acid, or equivalent
9. Stock and standard metal solutions
10. Whatman #42 filter paper
11. Glass or plastic funnels
12. Watch glasses
13. 30% Hydrogen peroxide

Reagent Preparation:

1. **Intermediate and working metal solutions:** Refer to the specific metal SOP for instructions on preparation.

2. **1:1 Hydrochloric acid (HCl):** Using a graduated cylinder, add 250 mL D.I. water to a to a 500 mL (or 1 L) repipettor. Carefully add 250 mL of concentrated HCl and mix.

3. **1:1 Nitric acid (HNO_3):** Using a graduated cylinder, add 250 mL D.I. water to a to a 500 mL (or 1 L) repipettor. Carefully add 250 mL of concentrated HNO_3 and mix.

Notes:

1. A separate digestion is required for mercury analyzed by the AA-Cold Vapor technique. (See "Mercury Digestion-Aqueous Samples")

2. All samples, duplicates, and spikes, as well as any required prep or digested blanks and standards, must be carried through the digestion procedure.

3. If samples boil or go to dryness (**any** dry spots on the bottom of the beaker) at any time during the digestion, some of the analyte may have been lost. The digestion must be discarded and the affected samples must be reprepared.

4. If elevated analyte levels are expected, the spike concentration may be increased accordingly.

Procedure:

Digestion Procedure for Flame-AA and ICP:

1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with D.I. water prior to use.

2. Measure out 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.

3. Add 2.0 mL of 1:1 HNO_3 and 10 mL of 1:1 HCl.

4. Cover with a watch glass and heat on the hot plate for 2 hours or until the volume has been reduced to between 25 and 50 mL. Adjust the temperature of the hot plate as needed to prevent samples from boiling.

5. Allow samples to cool. If any insoluble material remains, filter samples through Whatman #42 filters. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Rinse beakers and filters with D.I. water and dilute to volume to 100 mL.

6. Samples are now ready for analysis using the AA-flame or ICP methods.

Digestion Procedure for GFAA:

1. All glassware must be acid-washed with 1:1 nitric acid and thoroughly rinsed with D.I. water prior to use.

2. Measure out 100 mL aliquots of samples, blanks, and standards into 250 mL beakers using a graduated cylinder.

3. Add 1.0 mL of 1:1 HNO_3 and 2.0 mL of 30% H_2O_2.

4. Cover with a watch glass and heat on the hot plate for 2 hours or until the volume has been reduced to between 25 and 50 mL. Adjust the temperature of the hot plate as needed to prevent samples from boiling.

5. Allow samples to cool. If any insoluble material remains, filter samples through Whatman #42 filters. Quantitatively transfer digested samples, blanks, and standards into 100 mL volumetric flasks. Rinse beakers and filters with D.I. water and dilute to volume to 100 mL.

6. Samples are now ready for GFAA analysis.

Quality Control:

1. A digested blank and standard (spiked blank) must be included with each batch of samples that is digested. The blank is a check for possible contamination during the digestion process; the standard is a check for possible analyte loss during digestion.

2. A matrix spike and duplicate must be prepared, at a minimum, for every 10 samples digested. If fewer than 10 samples are digested a spike and duplicate are still required.

Effective Date: 7-2-91

ANTIMONY - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 204.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.005 mg/L

Optimum Range: 0.005 - 0.100 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	14
Slit Width (nm):	0.2
Slit Height:	Normal
Wavelength (nm):	217.6
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	2.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	1.40

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	85	5.0	3.0	NORMAL	NO
2	95	25.0	3.0	NORMAL	NO
3	120	10.0	3.0	NORMAL	NO
4	120	5.0	3.0	NORMAL	NO
5	900	10.0	3.0	NORMAL	NO
6	900	5.0	3.0	NORMAL	NO
7	900	2.0	0.0	NORMAL	NO
8	2300	1.0	0.0	NORMAL	YES
9	2300	2.0	0.0	NORMAL	YES
10	2300	2.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix Modifier Volume: 5 uL (0.25% Nickel Nitrate).

Calibration standards: 25.0, 50.0, 100.0 ug/L.

Graphite Tube Type: Pyrolytic coated partition tube.

Sample Handling: Acidify with nitric acid to pH <2. Analyze within 6 months.

Reagent Preparation:

1. **Standard Antimony Solution (1000 ug/L Antimony):** Pipet 1.00 mL of the 1000 ppm stock antimony solution into a 1000 mL volumetric flask, add 0.5 mL HNO_3 and dilute to volume with D.I. water. Prepare fresh daily.

2. **Calibration standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Antimony Standard	Dilute to
25.0 ug/L	2.5 mL of 1000 ug/L Sb	100 mL
50.0 ug/L	5.0 mL of 1000 ug/L Sb	100 mL
100 ug/L	10 mL of 1000 ug/L Sb	100 mL

3. **Nickel Nitrate 0.25% Solution:** In a 100 mL volumetric flask dissolve 1.25g of $Ni(NO_3)_2 \cdot 6H_2O$ in D.I. water and dilute to 100 mL. Prepare fresh every 6 months.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of background correction is required.

4. The use of halide acids should be avoided.

5. Nickel nitrate is added as a matrix modifier to control interferences.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

If antimony is to be analyzed in the concentration mode, use the 25.0, 50.0, and 100 ug/l standards for instrument calibration and follow the procedure for analysis in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable range(90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required. (See the Furnace Decision Tree for more detail.)

6. An EPA reference standard will be analyzed with each analysis.

Calculations:

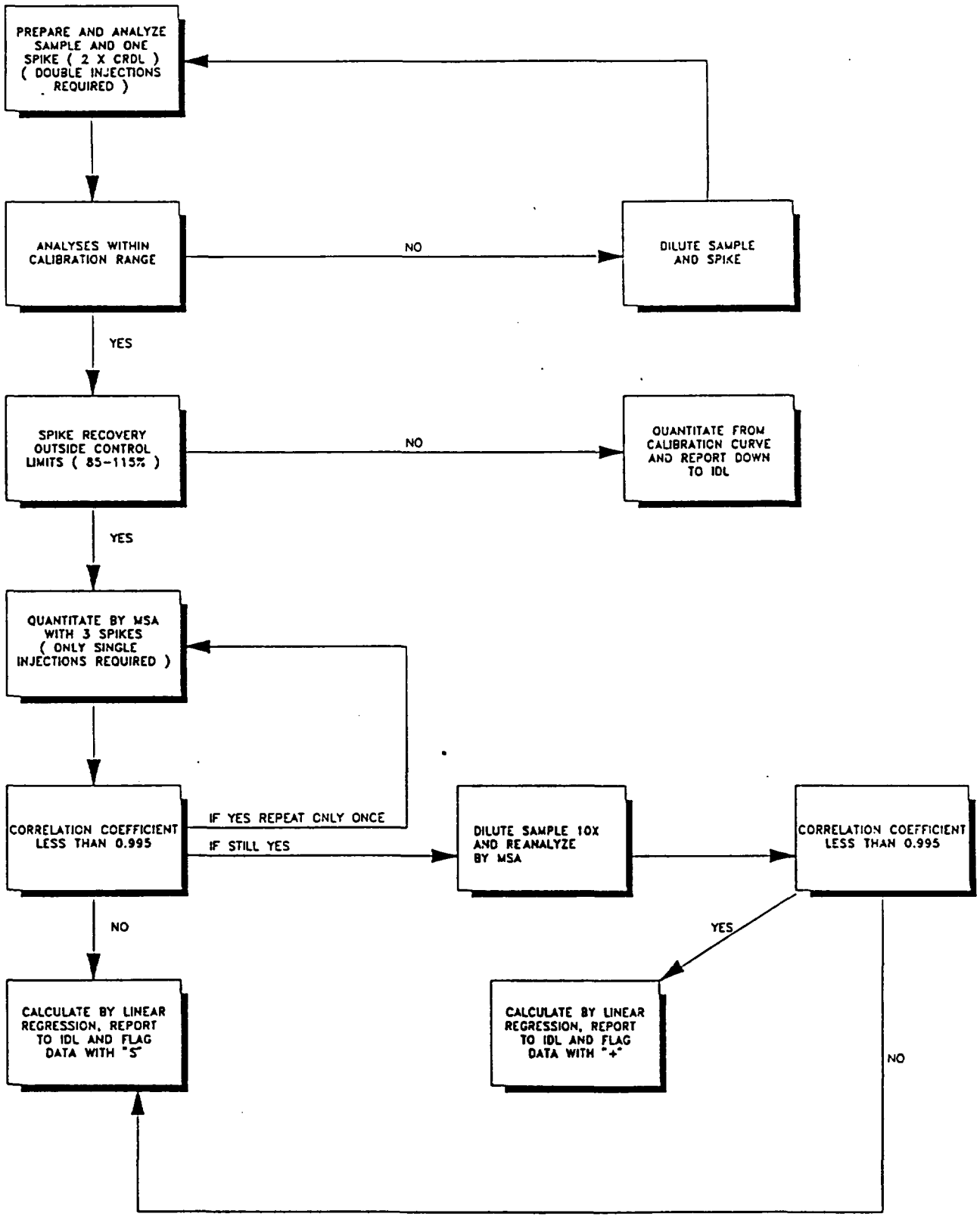
1. Calculate using the instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-298]

WARZYN

FURNACE CLP DECISION TREE



Effective Date: 7-2-91

ARSENIC - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 206.2

"Analytical Methods for Zeeman Graphite Tube Atomizers"-Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Sample Handling: Acidify with nitric acid to pH <2. Analyzed within 6 months. All samples must be digested prior to analysis.

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	10
Slit Width (nm):	1.0
Slit Height:	Normal
Wavelength (nm):	193.7
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	0.95

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	220	5.0	3.0	NORMAL	NO
3	240	40.0	3.0	NORMAL	NO
4	240	5.0	3.0	NORMAL	NO
5	1400	5.0	3.0	NORMAL	NO
6	1400	10.0	3.0	NORMAL	NO
7	1400	1.0	0.0	NORMAL	NO
8	2600	0.8	0.0	NORMAL	YES
9	2600	2.0	0.0	NORMAL	YES
10	2600	1.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix modifier volume: 5 uL (0.25% nickel nitrate).

Calibration standards: 10.00, 20.00, 50.00 ug/L.

Graphite Tube Type: Pyrolytic coated plateau tube

Reagent Preparation:

1. **Standard Arsenic Solution (1000 ug/L Arsenic):** Pipet 1.00 mL of the 1000 ppm stock arsenic solution into a 1000 mL volumetric flask, add 0.5 mL HNO_3 and dilute to the mark with deionized water. Prepare fresh monthly.

2. **Calibration standards:** Digest according to the appropriate digestion procedure. Prepare fresh monthly.

Concentration of Standard	Volume of Arsenic Standard	Dilute to
0 ug/L	0 mL of 1000 ug/L As	100 mL
10 ug/L	1 mL of 1000 ug/L As	100 mL
20 ug/L	2 mL of 1000 ug/L As	100 mL
50 ug/L	5 mL of 1000 ug/L As	100 mL

3. Nickel Nitrate (0.25%): In a 100 mL volumetric flask dissolve 1.25 g of $Ni(NO_3)_2 \cdot 6H_2O$ in D.I. water and dilute to 100 mL. Prepare fresh every 6 months.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. Nickel nitrate is added as a matrix modifier to minimize volatilization losses during the drying and charring steps.

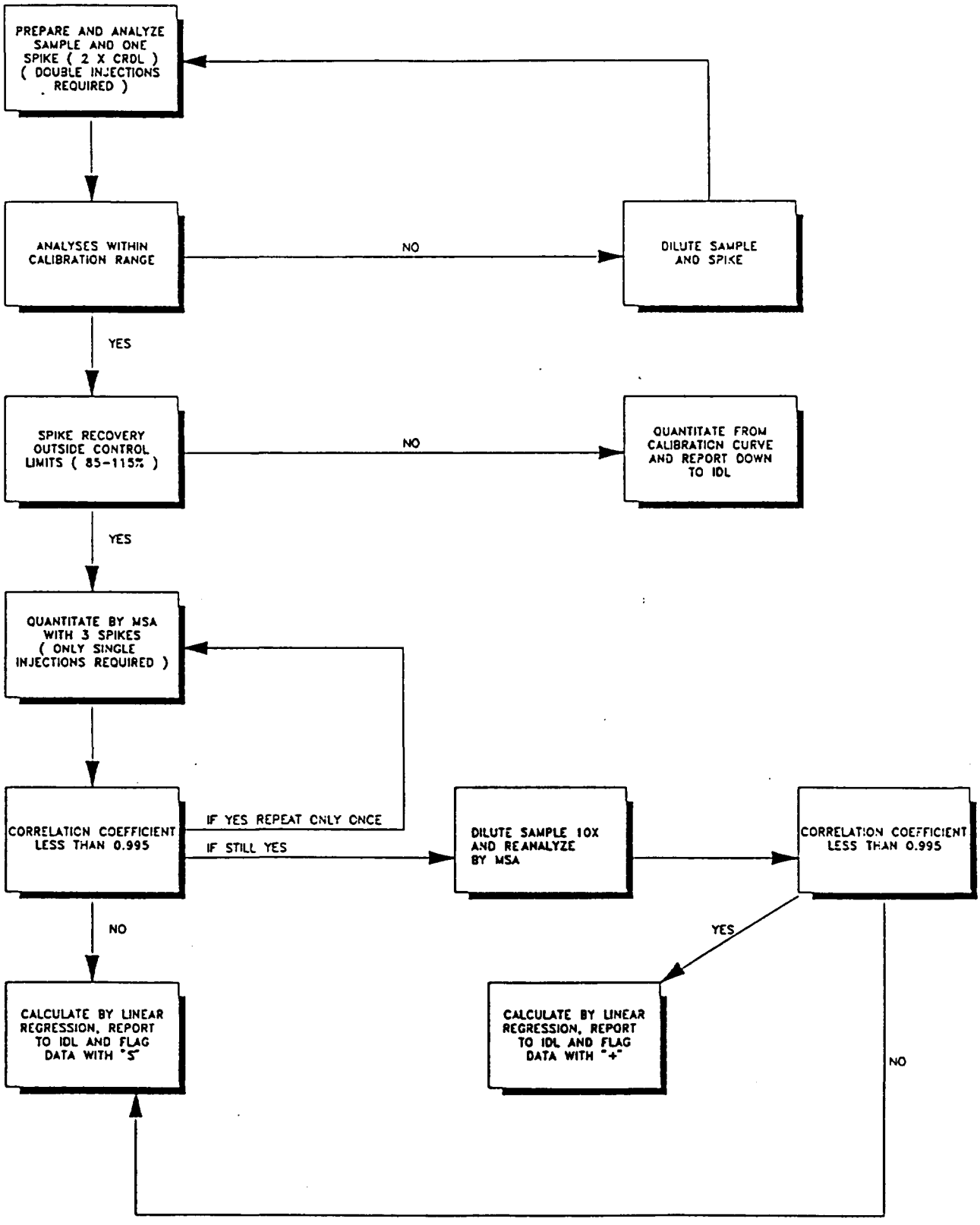
4. The use of background correction is required.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

If Arsenic is to be analyzed in concentration mode, use the 10.0, 20.0, and 50.0 ug/L standards for instrument calibration, and follow the procedures for analysis in the concentration mode.

WARZYN

FURNACE CLP DECISION TREE



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Effective Date: 7-2-91

CADMIUM - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 213.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.0002 mg/L

Optimum Range: 0.0002 - 0.0030 mg/L

Sample Handling: Acidify with nitric acid to pH < 2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	3
Slit Width (nm):	0.5
Slit Height:	Normal
Wavelength (nm):	228.8
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	0.70

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	230	5.0	3.0	NORMAL	NO
3	260	40.0	3.0	NORMAL	NO
4	260	5.0	3.0	NORMAL	NO
5	700	5.0	3.0	NORMAL	NO
6	700	5.0	3.0	NORMAL	NO
7	700	1.0	0.0	NORMAL	NO
8	2000	0.8	0.0	NORMAL	YES
9	2000	2.0	0.0	NORMAL	YES
10	2000	2.0	3.0	NORMAL	NO

Sample Volume: 12 uL

Matrix Modifier Volume: 4 uL (Monobasic ammonium phosphate)

Calibration standards: 1.00, 2.00, 3.00 ug/L

Graphite Tube Type: Pyrolytic coated plateau tube

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard Cadmium Solution (1000 ug/L Cadmium):** Pipet 1.00 mL of the 1000 ppm stock cadmium solution into a 1000 mL volumetric flask, add 1/2 mL HNO_3, and dilute to the mark with D.I. water. Prepare fresh daily.

2. **Working Cadmium Solution (100 ug/L Cadmium):** Pipet 10 mL of the 1000 ug/L cadmium into a 100 mL volumetric flask and dilute to the mark with D.I. water. Prepare fresh daily.

3. Standards (Prepare fresh daily.):

Concentration of Standard	Volume of Cadmium Standard	Dilute to
1.00 ug/L	1 mL of 100 ug/L Cd	100 mL
2.00 ug/L	2 mL of 100 ug/L Cd	100 mL
3.00 ug/L	3 mL of 100 ug/L Cd	100 mL

4. **Monobasic Ammonium Phosphate Solution (5000 mg/L):** Add 1.0 g of ammonium phosphate (monobasic) to a 100 mL volumetric flask. Dissolve in D.I. water and dilute to volume.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of background correction is required.

4. The cadmium flame or ICP procedure is recommended where concentrations are greater than 0.10 mg/L.

5. Ammonium phosphate is added as a matrix modifier to improve peak shape and allow higher ashing temperatures.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

Use of peak area is required.

If cadmium is to be analyzed in concentration mode, use the 1.00, 2.00, and 3.00 ug/L standards for instrument calibration and follow the procedures for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable range (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference sample will be analyzed with each analysis.

Calculations:

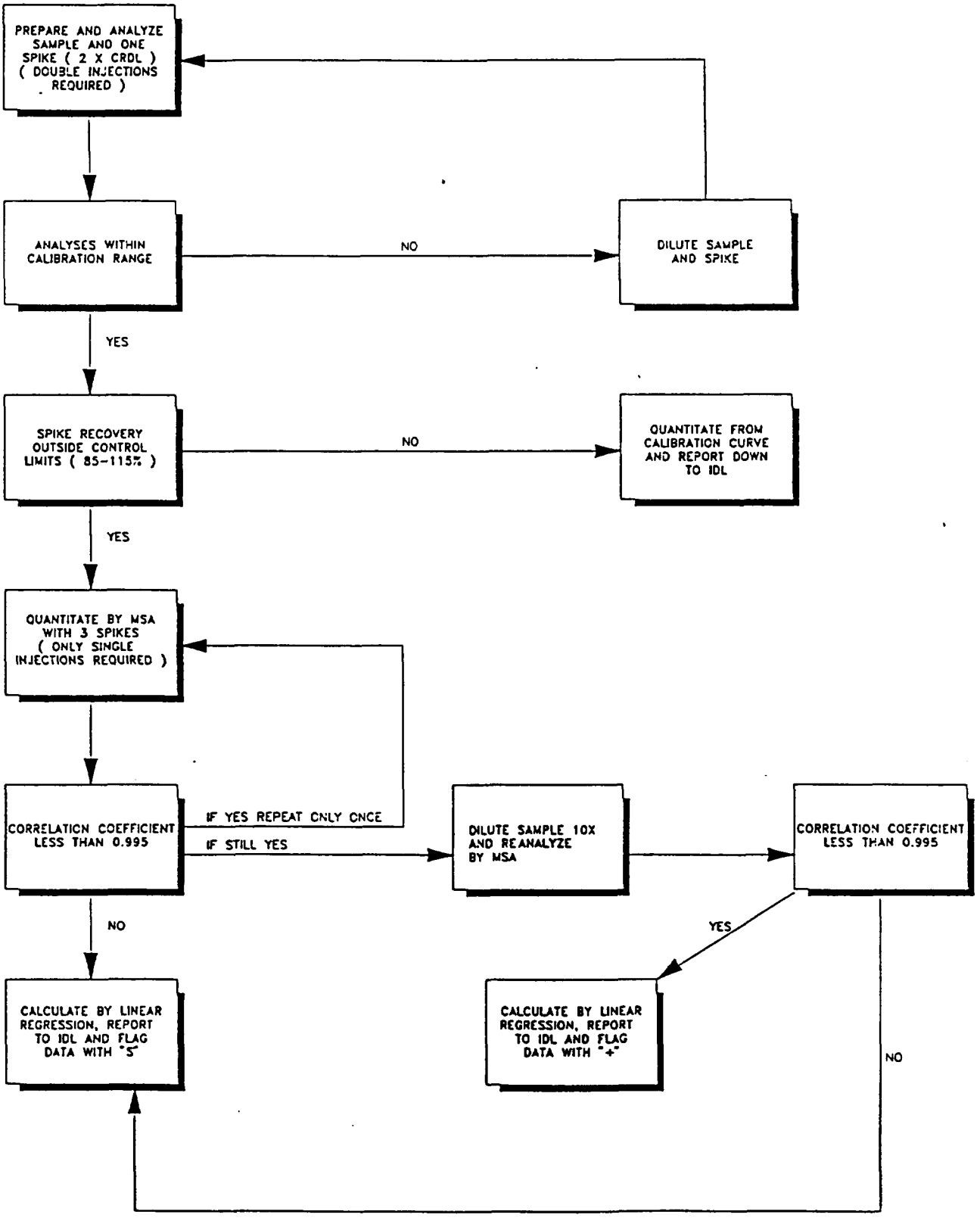
1. Calculate using instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-292]

WARZYN

FURNACE CLP DECISION TREE



Effective Date: 1-14-92

LEAD - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 239.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986.

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Sample Handling: Acidify with nitric acid to pH < 2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	4
Slit Width:	0.5
Slit Height:	Normal
Wavelength	283.3
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background:	On
Maximum Absorbance:	1.40

Furnace Parameters:

Step	Temp (*C)	Time (sec)	Gas Flow (L/Min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	220	5.0	3.0	NORMAL	NO
3	240	45.0	3.0	NORMAL	NO
4	240	5.0	3.0	NORMAL	NO
5	650	5.0	3.0	NORMAL	NO
6	650	15.0	3.0	NORMAL	NO
7	650	1.0	0.0	NORMAL	NO
8	2200	0.9	0.0	NORMAL	YES
9	2200	2.0	0.0	NORMAL	YES
10	2500	2.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix modifier volume: 5 uL 0.5% w/v Ammonium Phosphate Monobasic or 5 ul of lanthanum nitrate modifier.

Calibration standards: 3.0, 10.0, 20.0, 50.0 ug/L

Graphite Tube Type: Pyrolytic Coated Plateau Tube

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard lead solution (10.0 mg/L Lead):** Pipet 1.0 mL of the 1000 ppm stock lead solution into a 100 mL volumetric flask, add 0.5 mL HNO_3 and dilute to volume with deionized water. Prepare fresh daily.

2. **Standard lead solution (100ug/L Lead):** Pipet 1.0 mL of the 10.0 mg/L lead standard into a 100 mL volumetric flash, add 0.5 mL HNO_3 and dilute to volume with deionized water. Prepare fresh daily.

3. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Lead Standard	Dilute to
3.0 ug/L	3 mL of 100 ug/L Pb	100 mL
10.0 ug/L	10 mL of 100 ug/L Pb	100 mL
20.0 ug/L	20 mL of 100 ug/L Pb	100 mL
50.0 ug/L	50 mL of 100 ug/L Pb	100 mL

4. **Ammonium phosphate matrix modifier:** Dissolve 0.5g ammonium phosphate monobasic in 100mL D.I. water.

5. **Lanthanum nitrate matrix modifier:** Dissolve 5.864g of La_2O_3 in 10 ml concentrated nitric acid and dilute to 1 L with D.I. water.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of background correction is required.

4. Ammonium phosphate is added as a matrix modifier to improve peak shape and allow higher ashing temperatures. Ammonium phosphate is the preferred matrix modifier for groundwater, residential wells, and any other samples where chloride or sulfate concentrations are expected to be less than 100 mg/L. Due to its more corrosive nature, lanthanum nitrate should be used as matrix modifier only if chloride and/or sulfate concentrations are expected to exceed 100 mg/L.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

If lead is to be analyzed in the concentration mode, use the 3.0, 10.0, 20.0 and 50.0 ug/L standards for instrument calibration and follow the procedure for analyzing using the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard of 20.0 ug/L and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. **Criteria for standard additions are:**

   a. If the spike recovery is within 85 - 115%, standard additions are not required.

   b. If the spike recovery is outside 85 - 115%, standard additions are required. (**See Furnace Decision Tree for more detail.**)

6. An EPA reference sample will be analyzed with each analysis.

Calculations:

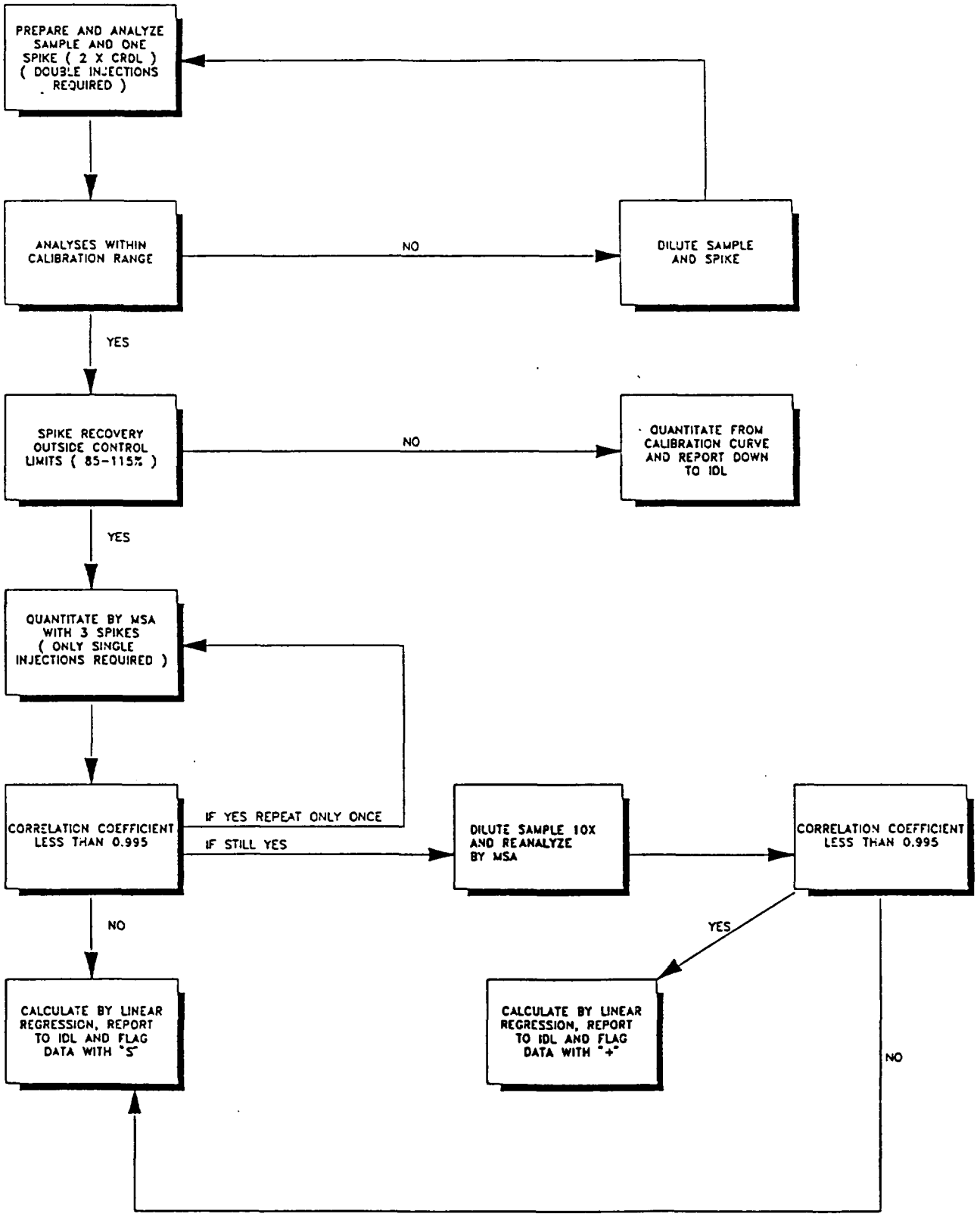
1. Calculate using the instrument concentration mode, or

3. For method of standard additions calculate using linear regression.

[rff-metcont-284]

WARZYN

FURNACE CLP DECISION TREE



Effective: 7-2-91

MERCURY DIGESTION LIQUID SAMPLES

Scope and Application: This mercury digestion method is applicable to drinking, surface, groundwater, domestic, and industrial wastewaters.

Method: Nitric/sulfuric acid digestion

Reference: EPA 1983, Method 245.1
"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Sample Handling: Preserve with concentrated HNO_3 to pH <2. Analyze within 28 days of sampling.

Reagents and Apparatus:

1. Water bath set @ 95°C
2. BOD bottles; 300 mL
3. Class A volumetric glassware
4. Instra-analyzed sulfuric acid
5. Instra-analyzed nitric acid
6. Potassium persulfate
7. Potassium permanganate
8. Sodium chloride
9. Hydroxylamine hydrochloride solution
10. Various Class A volumetric pipettes
11. Mercury stock and standard solutions

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. **Sodium chloride-hydroxylamine hydrochloride solution:** In a 1000ml volumetric flask dissolve 120.0 g of sodium chloride and 120.0 g of hydroxylamine hydrochloride in D.I. water, dilute to 1 liter.

2. **Potassium permanganate (5% solution, w/v):** In a 1000ml volumetric flask dissolve 50.0 g of potassium permanganate in D.I. water, dilute to 1 liter.

3. **Potassium persulfate (5% solution, w/v):** In a 1000ml volumetric flask dissolve 50.0 g of potassium persulfate in D.I. water, dilute to 1 liter.

4. **Intermediate mercury standard (10.0 mg/L):** Transfer 1.0 mL stock mercury (1000 mg/L) solution, plus 0.5 mL nitric acid, into a 100 mL volumetric flask and dilute to the mark with D.I. water. Prepare fresh daily!

5. **Working mercury standard (0.100 mg/L):** Transfer 1.0 mL of the 10.0 mg/L intermediate standard, plus 0.5 mL nitric acid, into a 100 mL volumetric flask and dilute to the mark with D.I. water. Prepare fresh daily!

Notes:

1. The mercury standards are volatile and unstable. Standards must be prepared daily.

2. Because of the toxic nature of mercury vapor, precaution must be taken to avoid inhalation. Vent the mercury vapor into an exhaust hood or pass the vapor through an absorbing media.

3. Hydroxylamine sulfate may be used rather than hydroxylamine hydrochloride.

4. All blanks, standards, and samples must be carried through the digestion procedure.

5. **Interferences:**

   a. Potassium permanganate is added to eliminate interferences from sulfide. Concentrations as high as 20 mg/L sulfide as sodium sulfide do not interfere.

   b. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on recovery of mercury from spiked samples.

   c. Seawaters, brines, and industrial effluents, high in chlorides, will require additional potassium permanganate. Care must be taken to ensure that the same amount of potassium permanganate is added to all samples, blanks, and standards so total volume remains constant.

Procedure:

All glassware is to be washed with soap and water, rinsed with tap water, acid rinsed with 10% HNO_3, and final rinsed with D.I. water.

Standard Preparation:

1. **The standard curve is to consist of the following standards:**

Standard Concentration
0.00 ug/L
0.50 ug/L
1.00 ug/L
5.00 ug/L
10.0 ug/L

2. Pipet 0, 0.5, 1.0, 5.0, and 10.0 mL aliquots of 0.10 mg/L working stock mercury solution to 300 mL BOD bottles.

3. Add D.I. water to bring volume to 100 mL and continue with the digestion procedure.

Sample Preparation:

1. Transfer 100 mL, or an aliquot diluted to 100 mL, to a 300 mL BOD bottle.

   To Spike: Pipette 1.0 mL of 0.10 mg/L mercury standard into the sample bottle.

Digestion:

1. Add 5 mL conc. sulfuric acid and 2.5 mL conc. nitric acid to each bottle. Mix by swirling.

2. Add 15 mL potassium permanganate solution to each bottle, mix by swirling. Allow to stand for at least 15 minutes. If the bottle does not remain purple in color, additional potassium permanganate is required. Equal volumes of potassium permanganate must be added to all bottles.

3. Add 8 mL of potassium persulfate solution to each bottle and heat for 2 hours in a water bath maintained at 95°C. Check the bottles periodically throughout the 2 hours to insure the samples remain purple. Add additional potassium permanganate, if needed, to all bottles in the digestion set.

4. Cool to room temperature.

5. Samples are now ready for analysis using the AA-cold vapor procedure.

Quality Control:

1. Refer to the cold vapor SOP for quality control requirements.

[rff-metcont-222]

Effective: 7-2-91

TOTAL MERCURY - AUTOMATED

Scope and Application: This method is applicable to digested drinking, surface, groundwater, domestic, and industrial wastewaters, soils, and sediments. All samples must be digested prior to analysis.

Method: Automated Cold Vapor

Reference: EPA 1984, Method 245.1, 245.5
SW846, 1982, Method 7471
"Vapor generation Accessory Operation Manual", Varian, 1984
"Statement of Work for Inorganic Analysis, No. 788", EPA 1989

Detection Limits: 0.20 ug/L

Optimum Range: 0.20-10.0 ug/L

Sample Handling: Samples should be kept capped until just prior to analysis.

Instrument Conditions:

Instrument Mode:		Absorbance
Calibration Mode:		Concentration
Measurement Mode:		Integration
Lamp Position:		1
Lamp Current (mA):		4
Slit Width (nm):	0.5	
Wavelength (nm):	253.7	
Flame:		Air only
Sample Introduction:		Auto Normal
Delay Time:		60
Time Constant:		0.05
Measurement Time (sec):		3.0
Replicates:		3
Background Correction:		On
Air Flow:	0.00	
Rinse Rate:		1
Rinse Time:		5.0
Recalibration Rate:		0
Reslope Rate:	0	

Reagents and Apparatus:

1. Varian SpectrAA20
2. Varian VGA-76 (cold vapor generator)
3. Varian PSC-56 (autosampler)
4. Sodium chloride
5. Hydroxylamine hydrochloride
6. Stannous chloride
7. Hydrochloric acid
8. Mercury lamp
9. Tygon tubing
10. Whatman #4 filter paper or equivalent

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. **Hydrochloric acid (20% v/v):** Add 100 mL of conc. HCl to 200 mL D.I. water in a 500 mL volumetric flask, dilute to 500 mL. **Prepare in the hood!**

2. **Stannous chloride (25% w/v):** Dissolve 125.0 g stannous chloride in 500 mL of 20% HCL. Prepare fresh every month.

3. **Sodium chloride-hydroxylamine hydrochloride solution:** Dissolve 120.0 g of sodium chloride and 120.0 g of hydroxylamine hydrochloride in D.I. water, dilute to 1 liter.

Notes:

1. Because of the toxic nature of mercury vapor, precaution must be taken to avoid inhalation. Vent the mercury vapor into an exhaust hood or pass the vapor through an absorbing media.

2. A 10% solution of stannous sulfate may be substituted for stannous chloride.

3. Hydroxylamine sulfate may be used rather than hydroxylamine hydrochloride.

4. **Interferences:**

   a. Potassium permanganate is added to eliminate interferences from sulfide. Concentrations as high as 20 mg/L sulfide as sodium sulfide do not interfere.

   b. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L have no effect on recovery of mercury from spiked samples.

   c. Seawaters, brines, and industrial effluents, high in chlorides, will require additional potassium permanganate. during the oxidation step, chlorides are converted to free chlorine which also absorbs at the same wavelength as mercury.

d. Certain volatile organic materials that absorb at this wavelength may also cause an interference. A preliminary run without reagents can determine if this type of interference is present.

5. Care must be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by leaving digested mercury samples uncapped in the hood for approximately 30 minutes after the addition of the sodium chloride hydroxylamine solution, or allowing a prepared autosampler tray to stand 10-20 minutes before starting an automated analytical run.

6. If particulates remaining in the digested sample cause obstructions in the autosampler tubing, samples can be filtered through Whatman #4 filter paper, or its equivalent, after excess permanganate has been reduced.

Procedure:

For instrument set-up procedures, refer to the Atomic Absorption Spectrometry, Flame section of this manual.

For concentration mode, use 0.5, 1.0, 5.0 and 10.0 ug/L standards to calibrate the instrument.

A. **Cold Vapor System Set-up:**

1. Insert quartz cell in burner chamber. (Attaches to the air/acetylene burner head.)

2. Visually align cell, checking light path with a white card or paper.

3. Select "Optimization" page and adjust cell for maximum signal.

4. Replace pump tubing on vapor generator.

5. Fill reagent bottles with D.I. water and 25% SnCl (stannous chloride) solution as labelled.

B. **Sample Analysis:**

1. Prior to analysis, add 6 mL of the sodium chloride-hydroxylamine solution to each bottle to reduce the excess permanganate. Additional sodium chloride-hydroxylamine may be needed to discharge the purple color; equal volumes must be added to all bottles in the digestion set.

2. Pour approximately 12 mL of samples, standards, and blanks into sample tubes and arrange on the autosampler.

3. Turn on argon supply (46 psi recommended).

4. Turn on autosampler power.

5. Turn on vapor generator power (peristaltic pump will run continuously while power is on. Check reagent levels periodically during long runs).

6. Allow pump to operate for 3 to 4 minutes to stabilize flow rates.

7. Start automatic run.

8. Run will stop automatically after it is completed. Press "stop" to release computer.

9. Pull tubing ends out of reagents and let pump empty the lines. Power off pump and release pump tubing. Turn off argon supply.

10. Power off autosampler, printer and AA if done with analyses for the day.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, tubing, lamp alignment, pump, etc.)

2. A quality control calibration check standard of 5.0 ug/L and a blank are to be analyzed initially and, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a check standard and a blank are still required. The last samples analyzed in the run are to be the check standard and blank. These standards must be within acceptable ranges (80-120%) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges or the data will be flagged appropriately.

4. An EPA or ERA reference standard will be analyzed with each analytical run. The reference standard must be within acceptable limits (80-120% of the true value) before any samples are analyzed.

Calculation:

1. Calculate using the instrument concentration mode, or

2. For method of standard additions, use linear regression.

[rff-metcont-225]

Effective: 7-2-91

POTASSIUM - VARIAN 20

Method: Flame Emission: Direct Aspiration

Reference: "Analytical Methods for Flame Spectrophotometry, Varian 1979.

"Standard Methods for the Examination of Water and Wastewater", 16th Edition, Method 322B, 1985.

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.10 mg/L

Optimum Range: 0.10 - 10.0 mg/L

Sample Handling: Acidify with nitric acid to pH <2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

1. Instrument mode: Emission
2. Wavelength: 766.5 nm
3. Slit Width: 1.0
4. Fuel: Acetylene
5. Oxidant: Air
6. Type of flame: Oxidizing, lean, blue
7. Standards to use for calibration: 0.50, 1.00, 2.00, 5.00, 10.0 mg/L.

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard Potassium Solution (100 mg/L Potassium):** Pipet 10 mL of the 1000 ppm stock potassium solution into a 100 mL volumetric flask, add 0.5 mL HNO_3, and dilute to volume with D.I. water.

2. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Potassium Standard	Dilute to
0.50 mg/L	0.5 mL of 100 mg/L	100 mL
1.00 mg/L	1 mL of 100 mg/L	100 mL
2.00 mg/L	2 mL of 100 mg/L	100 mL
5.00 mg/L	5 mL of 100 mg/L	100 mL
10.0 mg/L	10 mL of 100 mg/L	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Flame - Direct Aspiration section of this manual.

If potassium is to be analyzed in concentration mode, use the 1.00, 5.00, and 10.0 mg/L standards to calibrate the instrument and follow the procedure for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The emission readings should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, flame head alignment, etc.).

2. A quality control calibration standard of 1.00 mg/L and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

4. An EPA reference sample will be analyzed with each analysis.

Calculations:

1. Calculate using the instrument concentration mode, or

2. For method of standard additions, use linear regression.

[rff-metcont-280]

Effective: 7-2-91

SELENIUM - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 270.2

"Analytical Methods for Zeeman Graphite Tube Atomizer"-Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Height
Lamp Current (mA):	18
Slit Width (nm):	1.0
Slit Height:	Normal
Wavelength (nm):	196.0
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	1.20

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	220	5.0	3.0	NORMAL	NO
3	240	40.0	3.0	NORMAL	NO
4	240	5.0	3.0	NORMAL	NO
5	1400	5.0	3.0	NORMAL	NO
6	1400	10.0	3.0	NORMAL	NO
7	1400	1.0	0.0	NORMAL	NO
8	2600	0.8	0.0	NORMAL	YES
9	2600	2.0	0.0	NORMAL	YES
10	2600	1.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix Modifier Volume: 5 uL (0.25% nickel nitrate)

Standards to use for curve set-up: 5.0, 10.0, 20.0, 50.0 ug/L.

Graphite Tube Type: Pyrolytic coated plateau tube

Sample Handling: Acidify with nitric acid to pH < 2. Analyze within 6 months.

Reagent Preparation:

1. **Standard selenium solution (1000 ug/L Selenium):** Pipet 1.00 mL of the 1000 ppm stock selenium solution into a 1000 mL volumetric flask, add 0.5 mL HNO_3 and dilute to volume with D.I. Prepare fresh daily.

2. **Calibration standards:** Digest standards according to the appropriate digestion procedure. Prepare fresh monthly.

Concentration of Standard	Volume of Selenium Standard	Dilute to
5.0 ug/L	0.5 mL of 1000 ug/L Se	100 mL
10.0 ug/L	1 mL of 1000 ug/L Se	100 mL
20.0 ug/L	2 mL of 1000 ug/L Se	100 mL
50.0 ug/L	5 mL of 1000 ug/L Se	100 mL

3. **Nickel Nitrate (0.25%):** In a 100 mL volumetric flask dissolve 1.25 g of $Ni(NO_3)_2 \cdot 6H_2O$ in D.I. water and dilute to 100 mL. Prepare fresh every 6 months.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Chloride (> 800 mg/L) and sulfate (> 200 mg/L) interfere with this selenium procedure. Nickel nitrate is added as a matrix modifier to minimize these interferences.

3. Background correction is required.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

For concentration mode, use the 5.0, 10.0, 20.0 and 50.0 mg/L standards for instrument calibration and follow the procedure for analyzing using the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. Criteria for standard additions are:

   - If the spike recovery is within 85 - 115%, standard additions are not required.

   - If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference standard will be analyzed with each analysis.

Calculations:

1. Calculate using the instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-279]

Effective: 7-2-91

SILVER - VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 272.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.0005 mg/L

Optimum Range: 0.0005 - 0.010 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Height
Lamp Position:	7
Lamp Current (mA):	4
Slit Width (nm):	0.5
Slit Height:	Normal
Wavelength (nm):	328.1
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	1.30

FURNACE PARAMETERS

Step	Temp (*C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	85	5.0	3.0	NORMAL	NO
2	95	40.0	3.0	NORMAL	NO
3	120	10.0	3.0	NORMAL	NO
4	400	5.0	3.0	NORMAL	NO
5	400	1.0	3.0	NORMAL	NO
6	400	2.0	0.0	NORMAL	NO
7	2000	0.9	0.0	NORMAL	YES
8	2000	2.0	0.0	NORMAL	YES
9	2000	2.0	3.0	NORMAL	NO

Graphite Tube Type: Pyrolytic coated partition tube

Sample Volume: 20 uL

Standards to use for curve set-up: 1.00, 4.00, 10.0 ug/L

Sample Handling: Acidify with nitric acid to pH < 2. Analyze within 6 months.

Reagent Preparation:

1. **Standard Silver Solution (1000 ug/L Silver):** Pipet 1.00 mL of the 1000 ppm stock silver solution into a 1000 mL volumetric flask, add 5.0 mL HNO_3 and dilute to the mark with D.I. Prepare fresh daily.

2. **Working Silver Standard (100 ug/L Silver):** Pipet 10 mL of the 1000 ug/L silver standard into a 100 mL volumetric flask and dilute to the mark with D.I. Prepare fresh daily.

3. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Silver Standard	Dilute to
1.00 ug/L	1 mL of 100 ug/L Ag	100 mL
4.00 ug/L	4 mL of 100 ug/L Ag	100 mL
10.0 ug/L	10 mL of 100 ug/L Ag	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. Background correction is required.

4. The use of halide acids should be avoided.

5. Silver standards are light sensitive and tend to plate out on the container walls. Silver standards should be stored in amber glass bottles rather than plastic.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

For concentration mode, use the 1.0, 4.0 and 10.0 standards and follow the procedure for analyzing using the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within acceptable ranges (90-110% of the true value) or the samples run after the last acceptable calibration standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. **Criteria for standard additions are:**

   - If the spike recovery is within 85 - 115%, standard additions are not required.

   - If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference standard will be analyzed with each analysis.

Calculations:

1. Calculate using instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-277]

Effective: 7-2-91

SODIUM - VARIAN 20

Method: Flame Emission: Direct Aspiration

Reference: "Standard Methods for the Examination of Water and Wastewater", 16th Edition, Method 325B, 1985

"Analytical Methods for Flame Spectrophotometry", Varian, 1979

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 1.0 mg/L

Optimum Range: 1.0 - 100 mg/L

Sample Handling: Acidify with nitric acid to pH <2. Drinking waters and filtered groundwater free of particulate matter and organics may be analyzed directly, while wastewaters, leachates, solids, etc. must be digested prior to analysis (refer to appropriate digestion procedures). Analyze within 6 months.

Instrument Conditions:

1. Set signal to emission. (No lamp is required.)
2. Wavelength: 589.0 nm
3. Slit Width: 0.2 Normal
4. Fuel: Acetylene
5. Oxidant: Air
6. Type of flame: Oxidizing, lean, blue
7. Standards to use for curve set-up: 1.0, 5.0, 10.0, 25.0, 50.0, 75.0, 100.0 mg/L.

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard Sodium Solution (100 mg/L Sodium):** Pipet 10 mL of the 1000 ppm stock sodium solution into a 100 mL volumetric flask, add 1/2 mL HNO_3, and dilute to the mark with D.I. water.

2. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Sodium Standard	Dilute to
1.0 mg/L	1 mL of 100 mg/L Na	100 mL
5.0 mg/L	5 mL of 100 mg/L Na	100 mL
10.0 mg/L	1 mL of 1000 mg/L Na	100 mL
25.0 mg/L	2.5 mL of 1000 mg/L Na	100 mL
50.0 mg/L	5 mL of 1000 mg/L Na	100 mL
75.0 mg/L	7.5 mL of 1000 mg/L Na	100 mL
100.0 mg/L	10 mL of 1000 mg/L Na	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Flame - Direct Aspiration section of this manual but make the following changes:

1. Turn the burner head counter clockwise as far as it will go (approximately a 45° angle).

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The emission readings should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, flame head alignment, etc.).

2. A quality control calibration standard of 25.0 mg/L and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges or the samples run after the last acceptable check standard are to be reanalyzed.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

4. An EPA reference sample will be analyzed with each analysis.

Calculations:

1. Calculate using instrument concentration mode, or

2. For method of standard additions, use linear regression.

[rff-metcont-276]

Effective: 1-14-92

THALLIUM - 400 VARIAN

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 279.2

"Analytical Methods for Zeeman Graphic Tube Atomizers" - Varian 1986.

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	10
Slit Width (nm):	0.5
Slit Height:	Normal
Wavelength (nm):	276.8
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	0.55

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	125	5.0	3.0	NORMAL	NO
2	240	40.0	3.0	NORMAL	NO
3	240	10.0	3.0	NORMAL	NO
4	500	5.0	3.0	NORMAL	NO
5	500	10.0	3.0	NORMAL	NO
6	500	1.0	0.0	NORMAL	NO
7	2400	1.0	0.0	NORMAL	YES
8	2400	2.0	0.0	NORMAL	YES
9	2400	1.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Matrix Modifier Volume: 5 uL (1% H_2SO_4)

Calibration standards: 10.00, 25.00, 50.00 ug/L.

Graphite Tube Type: Pyrolytic coated plateau tube

Sample Handling: Acidify with nitric acid to pH <2. Analyze within 6 months.

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard Thallium Solution (1000 ug/L Thallium):** Pipet 1.00 mL of the 1000 ppm stock thallium solution into a 1000 mL volumetric flask, add 0.5 mL HNO_3 and dilute to volume with D.I. water. Prepare fresh every month.

2. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Thallium Standard	Dilute to
10.0 ug/L	1.0 mL of 1000 ug/L Tl	100 mL
25.0 ug/L	2.5 mL of 1000 ug/L Tl	100 mL
50.0 ug/L	5.0 mL of 1000 ug/L Tl	100 mL

3. H_2SO_4 (1%): Add 1.0 mL of concentrated H_2SO_4 to 90 mL D.I. water.Dilute to 100 mL.

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. 1% H_2SO_4 is added as a matrix modifier.

4. The use of background correction is required.

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Injection section of this manual.

For the use of the concentration mode, use the 10.0, 25.0 and 50.0 mg/L standards for instrument calibration and follow the procedure for analyzing in the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, furnace alignment, lamp alignment, graphite tube, etc.).

2. A quality control calibration standard and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. **Criteria for standard additions are:**

   - If the spike recovery is within 85 - 115%, standard additions are not required.

   - If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference standard will be analyzed with each analysis.

Calculations:

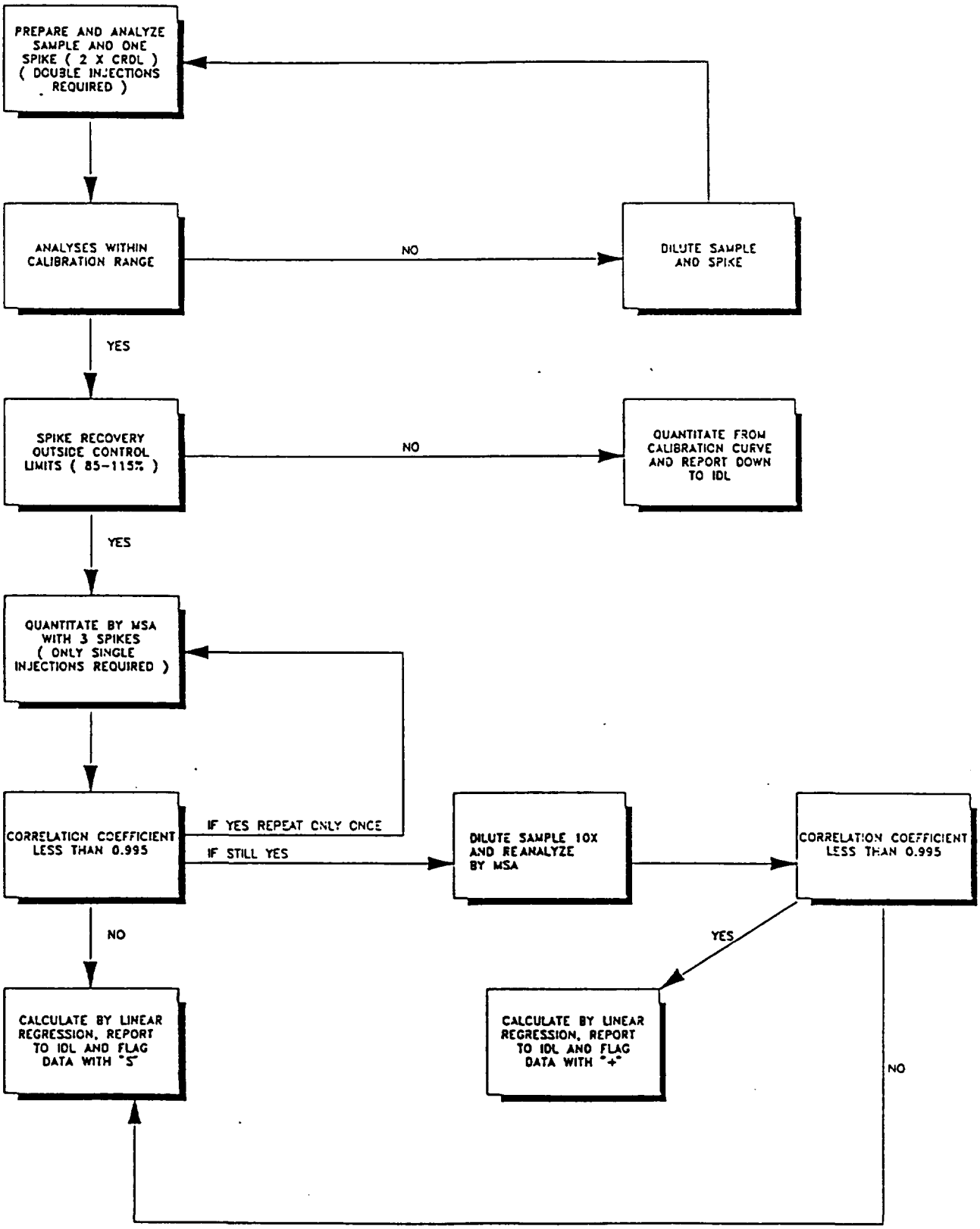
1. Calculate using instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-274]

WARZYN

FURNACE CLP DECISION TREE



Effective: 7-2-91

VANADIUM-VARIAN 400

Method: AA - Furnace; Direct Injection

Reference: EPA 1984, Method 286.2

"Analytical Methods for Zeeman Graphite Tube Atomizers", Varian, 1986

"Statement of Work for Inorganic Analysis", ILM01.0, EPA 1990

Detection Limit: 0.002 mg/L

Optimum Range: 0.002 - 0.050 mg/L

Instrument Conditions:

Instrument Mode:	Absorbance
Calibration Mode:	Concentration
Measurement Mode:	Peak Area
Lamp Current (mA):	10
Slit Width (nm):	0.2
Slit Height:	Normal
Wavelength (nm):	318.5
Sample Introduction:	Sampler Premixed
Time Constant:	0.05
Measurement Time (sec):	1.0
Replicates:	2
Background Correction:	On
Maximum Absorbance:	1.80

FURNACE PARAMETERS

Step	Temp (°C)	Time (sec)	Gas Flow (L/min)	Gas Type	Read Command
1	95	5.0	3.0	NORMAL	NO
2	130	40.0	3.0	NORMAL	NO
3	1400	10.0	3.0	NORMAL	NO
4	1400	10.0	3.0	NORMAL	NO
5	1400	1.0	0.0	NORMAL	NO
6	2700	0.7	0.0	NORMAL	YES
7	2700	2.0	0.0	NORMAL	YES
8	2700	2.0	3.0	NORMAL	NO

Sample Volume: 20 uL

Standards to use for curve set-up: 10.0, 20.0, 50.0 ug/L.

Graphite Tube Type: Pyrolytic coated partition tube

Sample Handling: Acidify with nitric acid to pH <2. Analyze within 6 months.

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Standard vanadium solution (1000 ug/L vanadium):** Pipet 1.0 mL of the 1000 ppm stock vanadium solution into a 1000 mL volumetric flask, add 1/2 mL HNO_3 and dilute to the mark with deionized water. Prepare fresh daily.

2. **Standards:** (Prepare fresh daily.)

Concentration of Standard	Volume of Vanadium Standard	Dilute to
10.0 ug/L	1.0 mL of 1000 ug/L V	100 mL
20.0 ug/L	2.0 mL of 1000 ug/L V	100 mL
50.0 ug/L	5.0 mL of 1000 ug/L V	100 mL

Notes:

1. Samples must be diluted to obtain concentrations within the optimum concentration range.

2. Standards are to be prepared in the same acid concentrations as the samples being analyzed.

3. The use of background correction is required.

4. The use of halide acids should be avoided.

5. Vanadium is a refactory metal, extra care should be taken that sample is not boiled during the digestion (vanadium is easily lost).

Procedure: For the analysis procedure, refer to the Atomic Absorption Spectrometry, Furnace - Direct Aspiration section of this manual.

For the use of concentration mode, use the 10.0, 20.0 and 50.0 standards and follow the procedure for using the concentration mode.

Quality Control:

1. Establish a standard curve with the standards listed above plus a blank. Record the absorbance check standard in the absorbance check book. The absorbances should remain consistent from run to run. If not, necessary troubleshooting must be performed before continuing (check wavelength, flame head alignment, lamp alignment, etc.)

2. A quality control calibration standard of 20.0 ug/L and a blank are to be analyzed, initially and after every 10 samples. If less than 10 samples are analyzed, a calibration standard and blank are still required. The last samples analyzed in the run are to be the calibration standard and blank. These standards must be within the acceptable ranges (90-110% of the true value) or the samples run after the last acceptable check standard are to be reanalyzed.

3. Analyze a standard at, or less than, the contract required detection limit after the initial calibration verification and blank.

4. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges, or data must be flagged appropriately.

5. For every sample analyzed, an analytical spike (at the bench) must be run to verify that standard additions are not required. **Criteria for standard additions are:**

   - If the spike recovery is within 85 - 115%, standard additions are not required.

   - If the spike recovery is outside 85 - 115%, standard additions are required. (See Furnace Decision Tree for more detail.)

6. An EPA reference sample will be analyzed with each analysis.

Calculations:

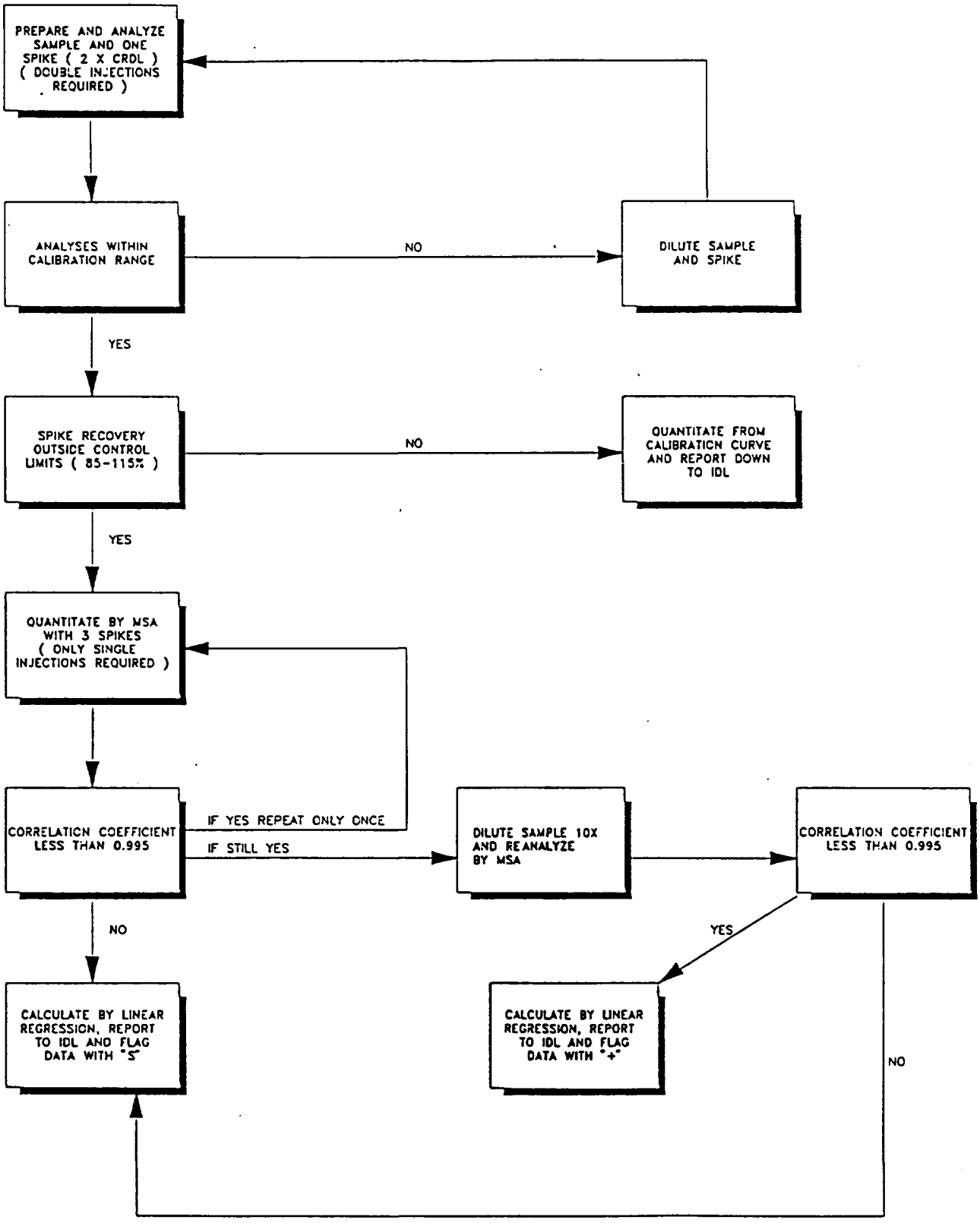
1. Calculate using instrument concentration mode, or

2. For method of standard additions calculate using linear regression.

[rff-metcont-272]

WARZYN

FURNACE CLP DECISION TREE



Effective Date: 5.24.91

CYANIDE, TOTAL - DISTILLATION

Scope and Application: This method is applicable to the determination of cyanide in drinking water, surface water, ground-water, sludges, soils and industrial wastes.

Methods: Distillation, Automated Colorimetric

Reference: EPA 1983, Method 335.2,
SW-846, Method 9012
Standard Methods, 16th Edition, Method 412

Detection Limit: 0.005 mg/L

Optimum Range: 0.005 - 0.400 mg/L

Sample Handling: Preserve with sodium hydroxide to pH > 12 and refrigerate at 4°C
Analyze samples within 12 days of receipt date.

Reagents and Apparatus:

1. Cyanide reflux distillation apparatus
2. 25 mL and 50 mL graduated cylinders
3. Vacuum pump
4. Heating mantle
5. 250 mL volumetric flasks
6. Sodium hydroxide
7. Sulfuric acid, concentrated
8. Magnesium chloride
9. Deionized water
10. Bismuth nitrate
11. Sulfamic acid
12. Acetic acid, concentrated
13. Sodium thiosulfate, crystals

Reagent Preparation: (Prepare fresh every 6 months, unless otherwise noted.)

1. **Sodium hydroxide (1.25N):** Dissolve 50.0 g NaOH in D.I. water and dilute to 1 liter in a volumetric flask. Store in a plastic bottle.

2. **Magnesium chloride solution:** Dissolve 510.0 g $MgCl_2 \cdot 6H_2O$ in D.I. water and dilute to 1 liter. Store in a plastic bottle.

3. **Stock cyanide solution (1000 mg/L):** Dissolve 0.6275 g KCN and 0.5 g KOH and dilute to 250 mls with D.I. water in a volumetric flask. Prepare fresh every month. **Caution: Toxic!** Refrigerate.

4. **Standard cyanide solution (5 mg/L):** Pipet 5 mL of stock cyanide solution into 1 L volumetric flask containing approximately 500 mL D.I. water and 2 mL of 10N NaOH as a preservative. Dilute to volume with DI water. **Prepare fresh daily.** Refrigerate.

5. **Bismuth nitrate solution:** Dissolve 30.0 g of $Bi(NO_3)_3$ in 100 mL of D.I. water. While stirring, add 250 mL of concentrated acetic acid. Stir until dissolved. Dilute to 1 liter with D.I. water.

6. **Sulfamic acid solution:** Dissolve 40.0 g of sulfamic acid in D.I. water. Dilute to 1 liter.

Notes:

1. **Caution:** Use care in handling of samples with cyanide because of the toxicity. Avoid skin contact, inhalation, or ingestion. **Always have a respirator on when doing this test.**

   If a sample begins to bump or back up the tube, quickly increase the flow rate, and turn the heat down (or off) until bumping subsides.

   If a sample does boil over, proceed as follows:

   - Pull inlet tube out
   - Turn heat off (For your protection, use gloves.)
   - Put sample and heating mantle into hood
   - When sample is cool remove from mantle and heat mantle in hood on high until acid fumes have dissipated.

2. Oxidizing agents, such as chlorine, interfere by decomposing cyanides. If chlorine is believed to present, put a drop of sample on potassium iodide starch paper. If paper turns bluish, add a few crystals of sodium thiosulfate ($Na_2S_2O_3$) to the sample, mix, and retest. Continue adding sodium thiosulfate until free from chlorine. Then, add 0.1 g sodium thiosulfate in excess.

3. Sulfides interfere by forming thiocyanate at a high pH. If sulfides are believed to be present, put a drop of sample on lead acetate test paper treated with acetic acid buffer solution at ph4. Darkening of paper indicates sulfides. If sulfides are present, add 50 mL of bismuth nitrate solution after the air rate is set through the air inlet tube. Mix for 3 minutes prior to addition of H_2SO_4.

   Alternatively, $Cd(NO_3)_2 \cdot 4H_20$, $CdCO_3$ or $PbCO_3$ can be added after the distillation, but prior to color development. Bismuth nitrate added prior to the distillation process is the preferred choice.

4. Fatty acids, high carbonates, and aldehydes can interfere. Refer to Standard Methods for troubleshooting.

5. High concentrations of NO_3 and NO_2 can give false positive results. If samples contain high concentrations of NO_3 and/or NO_2, add 50 mL of sulfamic acid solution after the air rate is set through the air inlet tube. Mix for 3 minutes prior to addition of H_2SO_4.

6. Do not use bismuth nitrate and sulfamic acid aon the same sapmle. Pretreatment with both results in poor (bias low) cyanide recovery.

Procedure:

1. All glassware is to be soap and water washed, tap rinsed, and deionized rinsed prior to analyses. Dichromate or acetone may also be used to clean the glassware prior to the soap and water wash.

2. Connect and set up cyanide reflux distillation apparatus as shown in Figure 2.

3. Prepare the 0.100 mg/L cyanide digestion standard as follows:

   Add 5 mL of the 5 mg/L cyanide solution to 250 mL of DI water. (Prepare in the distillation flask.)

4. Pour 250 mL of sample into cyanide distilling flask. If a solid or semi-solid sample is to be analyzed, use a 1.0 g sample size and add 250 mL of D.I. water to the distilling flask. (Record the amount of sample used.) Add an additional 250 mL D.I. water for a total volume of 500 mL in the distillation flask. Add 3-5 boiling chips.

   To Spike: Add 5 mL of the 5 mg/L cyanide solution to the 250 mL of sample for a final concentration of 0.100 mg/L.

5. Using a graduated cylinder, add 50 mL 1.25 N sodium hydroxide to the absorber tube and connect.

6. Turn on vacuum pump and adjust so that one bubble per second enters the distillation flask through the air inlet tube.

7. Slowly add 25 mL concentrated sulfuric acid through the air inlet tube. Rinse the tube with D.I. water and wait for about 2-3 minutes, until the sulfuric acid has been dispersed into the sample.

8. Using a graduated cylinder, add 20 mL magnesium chloride solution into the air inlet tube and rinse the tube with D.I. water.

9. Turn heating mantle on to setting of 10 till sample boils (approximatley 15 minutes). Watch vacuum rate carefully and adjust as necessary maintaining a rate of one bubble per second. As temperature increases, bubbling increases, and the solution can be drawn from the absorption tube or blown out the air inlet tube.

10. After sample starts boiling, turn heating mantle down to 6-7. **Watch vacuum rate carefully and adjust as necessary** maintaining a rate of one bubble per second. Reflux for one hour.

11. Turn off heat and continue vacuum for 15 minutes.

12. Remove inlet tubes.

13. Disconnect absorber, DI rinse absorber top into absorbing solution, and shut off vacuum pump.

14. Pour solution from absorber tube into a 250 mL volumetric flask. Using D.I. water, rinse the absorption tube (3 times) and add to the volumetric flask. Dilute to mark with DI water. Mix by inverting.

15. Distillates are ready for analysis. Proceed with Lachat SOP CNAAHC for the automated colorimetric step.

Quality Control:

1. The standard curve does not need to be carried through the distillation procedure.

2. A reagent blank is to be analyzed with each set of samples. This blank is to be carried through the distillation steps as a check for contamination.

3. A quality control distilled check standard of 0.100 mg/L cyanide is to be analyzed with each set of samples. This standard is to be carried through the entire procedure including the distillation step.

4. A known reference standard (LCS) is to be analyzed with each set of samples. This standard is to be carried through the entire procedure including the distillation steps. This standard must be within 80-120% of the true value and within 95% confidence limits (if available) or the samples are to be reanalyzed.

5. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicate results are to be within acceptable ranges.

6. Aqueous and solid/semi-solid samples are separate matrices. Duplicates and spikes are required for each matrix type.

Calculation:

1. Calculate distillate concentration with Lachat QuikChem software, in the concentration mode, using the IBM XT computer. (Be sure to calculate in any distillation dilution into the final result.)

$$\text{ug/L CN} = \frac{(\text{distillate volume,mL})(\text{distillate concentration,mg/L}) \times 1000}{(\text{sample volume,mL})}$$

$$\text{mg/kg CN} = \frac{(\text{distillate volume,mL})(\text{distillate concentration,mg/L})}{(\text{sample weight,gm})}$$

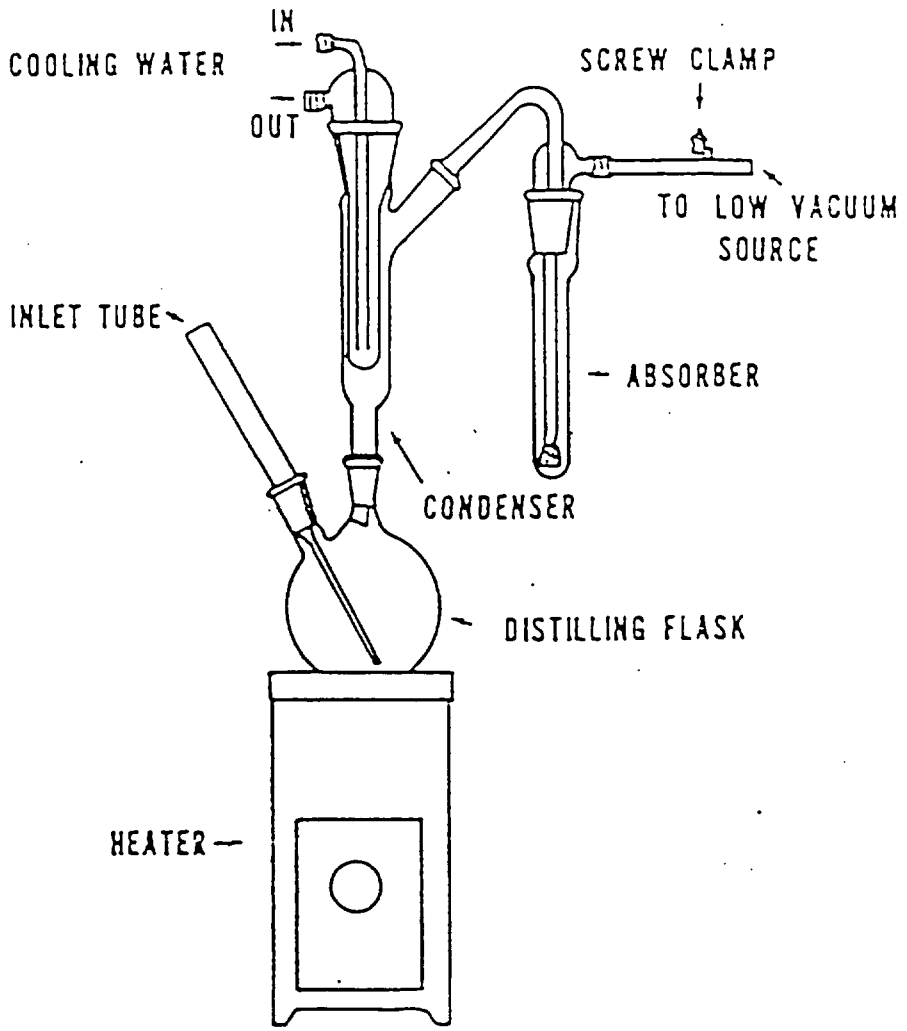


FIGURE 2

CYANIDE DISTILLATION APPARATUS

Effective Date: 5/24/91

TOTAL CYANIDE - AUTOANALYZER - (HEATED METHOD)

Scope and Application: This method is applicable to **distilled** groundwater, drinking water, wastewater, sediments and soils. All samples must be distilled prior to analysis with the autoanalyzer. (Refer to SOP # CNDISC.)

Reference: EPA, 1983, Method 335.3
Lachat Instruments, 1990, Method 10-204-00-1-A
Standard Methods, 16th Edition, pages 337-338
SW-846, Method 9012
SOW No. 788 including Rev 2/89 and 6/89

Instrument Detection Limit: 0.010 mg/L

Optimum Range: 0.005 - 0.400 mg/L

Sample Handling: Samples should be capped and refrigerated at 4°C after distillation. Samples must be analyzed within 3 days after distillation and within 12 days of receipt date.

Instrument Conditions:

1. Pump speed: 35
2. Cycle period: 50 seconds
3. Load period: 20 seconds
4. Inject period: 15 seconds
5. Inject to start of peak period: 30 seconds
6. Inject to end of peak period: 78 seconds
7. Gain: 420
8. Zero: 350
9. Interference filter: 570 mm
10. Sample loop: 150 cm (0.80 mm i.d.)
11. Standards for calibration: 0, 0.010, 0.020, 0.100, 0.200, 0.400 mg/L
12. Water Bath 45°C (Position A).

Reagent Preparation: (Prepare fresh every 6 months unless otherwise noted.)

1. **Degassed Milli-Q-water - 2 options:**

   a. Boil Milli-Q water vigorously for 5 minutes. Cool and store in cubitainer.

   b. Bubble helium, using the fritted gas dispersion tube, through 20 L Milli-Q water for 15-20 minutes. Store in cubitainer.

2. **Carrier - 0.25N NaOH:** In a 1 L volumetric flask, dissolve 10.0 g NaOH in 900 mL DI water. Dilute to the mark and invert several times. Filter through 0.45 micron filter paper. Store in a plastic bottle.

3. **Phosphate buffer - 0.71 M:** In a 1 L volumetric flask, dissolve 97.0 g anhydrous potassium dihydrogen phosphate (potassium phosphate, monobasic, anhydrous, KH_2PO_4) in 800 mL degassed MQ water. Add 8.1 mL concentrated (85%) phosphoric acid. Dilute to the mark with degassed MQ water and invert several times.

4. **Chloramine-T solution:** In a 500 ml volumetric dissolve 2.0 g of chloramine-T in degassed Milli-Q. Dilute to the mark and invert several times. Prepare fresh weekly and store refrigerated.

5. **Pyridine - barbituric acid reagent:** In the fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100 mL of degassed MQ water, rinsing down the sides of the beaker to wet the barbituric acid. Add 75 mL pyridine (C_5H_5N) while stirring with a stir bar. Mix until barbituric acid dissolves. Add 15 mL concentrated HCl and stir. Transfer to a 1 L volumetric flask, dilute to the mark with degassed MQ water and invert several times. Refrigerate. Prepare fresh every 2 months.

6. **Stock cyanide solution (1000 mg/L):** Dissolve 0.6275 g KCN and 0.5 g KOH and dilute to 250 mL with D.I. water in a volumetric flask. Prepare fresh every month. Refrigerate. **Caution: Toxic!**

7. **Standard cyanide solution (5.0 mg/L):** Pipet 5 mL of stock cyanide solution into 1 L volumetric flask, add approximately 500 mL DI water. Add 2 mL of 10N NaOH as a preservative and dilute to volume with DI water. Prepare fresh daily. Refrigerate.

8. **Cyanide standards:** Prepare by pipetting the volumes noted below into 250 mL volumetric flasks, adding 50 mL of 1.25N NaOH, and diluting to the mark with degassed MQ water. (The 1.25N NaOH must be added - very important!) **Prepare fresh daily.**

Concentration of Standard	Letter Identifier	Volume of 5.0 mg/L working standard (ml)	Dilute to
0.000 mg/L	A	0 mL	250 mL
0.010 mg/L	B	0.5 mL	250 mL
0.020 mg/L	C	1.0 mL	250 mL
0.100 mg/L	D	5.0 mL	250 mL
0.200 mg/L	E	10 mL	250 mL
0.400 mg/L	F	20 mL	250 mL

Note: Computer refers to standards by letter.

Notes:

1. This chemistry is temperature sensitive. The heated method reduces or eliminates sensitivity drift due to temperature changes.

2. Use wasteline coil to help eliminate air spikes.

3. Any sample dilutions must be diluted with 0.25N NaOH, **not** water. You may use the carrier or the zero standard for this.

4. Interferences are reduced or eliminated by the distillation procedure. Cyanide analyses suffer from many interferences. See EPA and Standard Methods references for detailed discussion. Information and recommendations for the manual pyridine-barbituric acid color development also apply to this automated method.

5. Samples must be diluted to obtain concentrations within the optimum working range.

6. The gain and zero settings are guidelines and must be optimized each day.

7. Color is an interference, dilute the sample and also manually spike the dilution to confirm the quality of the result.

System Operation:

1. Refer to "Auto Analyzer Operation Start-up Procedure" (IOP# LAAC-Section A).

2. Analyze an initial calibration check standard, a blank, a distilled known reference standard, a distilled standard and a distilled blank at the beginning of each run. The blanks must be below the detection limit and the standards must be within required control limits before any samples are analyzed.

3. Spikes are be distilled at a level of 0.100 mg/L.

4. The calibration check standard is 0.100 mg/L (D).

5. The distilled standard is 0.100 mg/L.

6. If a sample and spike are **overrange:**

   a. Dilute the sample and spike if dilution ≤ 1:5. The distilled spike should be detectable.
   b. Dilute the sample, spike and analyze a manual spike if dilution > 1:5.

7. Refer to Auto Analyzer shut-down procedure. (IOP# LAAC-Section B).

Quality Control:

1. Establish a standard curve with the standards listed above. The derived concentrations for each calibration standard must read within 10% of the true value. The derived value for the blank must be less than the method detection limit.

2. A quality control calibration check standard of 0.100 mg/L (D) and a blank are to be analyzed initially and at a minimum, after every 10 samples. If less than 10 samples are analyzed, a calibration check standard and blank are still required. The last samples analyzed in the run are to be the calibration check standard and blank. These standards must be within the acceptable ranges and blanks must be below the method detection limit or the samples run after the last acceptable calibration check standard and blank are to be reanalyzed.

3. Duplicate and spike a minimum of 1 out of 10 samples. If less than 10 samples are analyzed, a duplicate and spike are still required. Spike recoveries and duplicates are to be within acceptable ranges or data must be flagged appropriately. (These samples must be carried through the distillation step.)

Calculations:

1. Calculate with Lachat QuikChem software, in the concentration mode, using the IBM XT computer. Be sure to calculate any digestion dilution into the final result.